Development and test of new technologies for manufacturing high purity germanium segmented detector

Walter Raniero
INFN – LNL

mail: walter.raniero@lnl.infn.it

EUROPEAN LABORATORIES FOR ACCELERATOR BASED SCIENCES

Instrumentation and Training for Accelerators Based Nuclear Spectroscopy and Reaction Dynamics

Istituto Nazionale di Fisica Nucleare

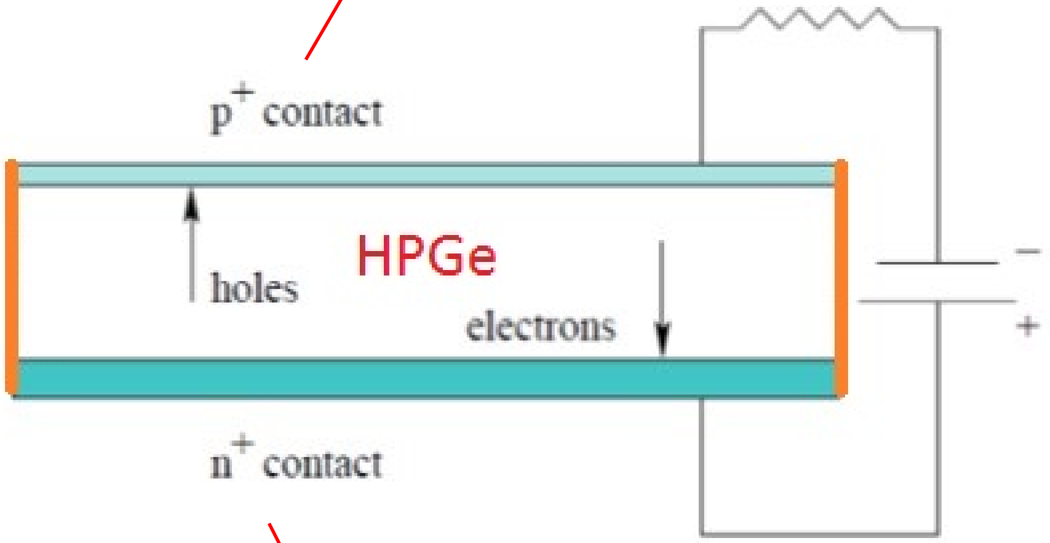
OUTLINE

- Introduction: HPGe gamma detectors
- Gamma detector state of the art
- *PLM (Pulse Laser Melting):* Next generation of segmented contact/junction on HPGe detectors
- PLM Planar gamma segmented detectors
- PLM Coxial gamma segmented detectors
- Neutron damage in PLM planar segmented detectors

Schematic HPGe planar gamma detector

B doped = **0.3 µm**
(implantation)

Surface passivation:



Li doped = **0.9 mm**
(diffusion)

HPGe
Charge carrier density: 10^{10} cm^{-3}

HPGe planar detector

Diameter: 40mm
Height: 20mm

HPGe gamma detector (chemical Passivation)

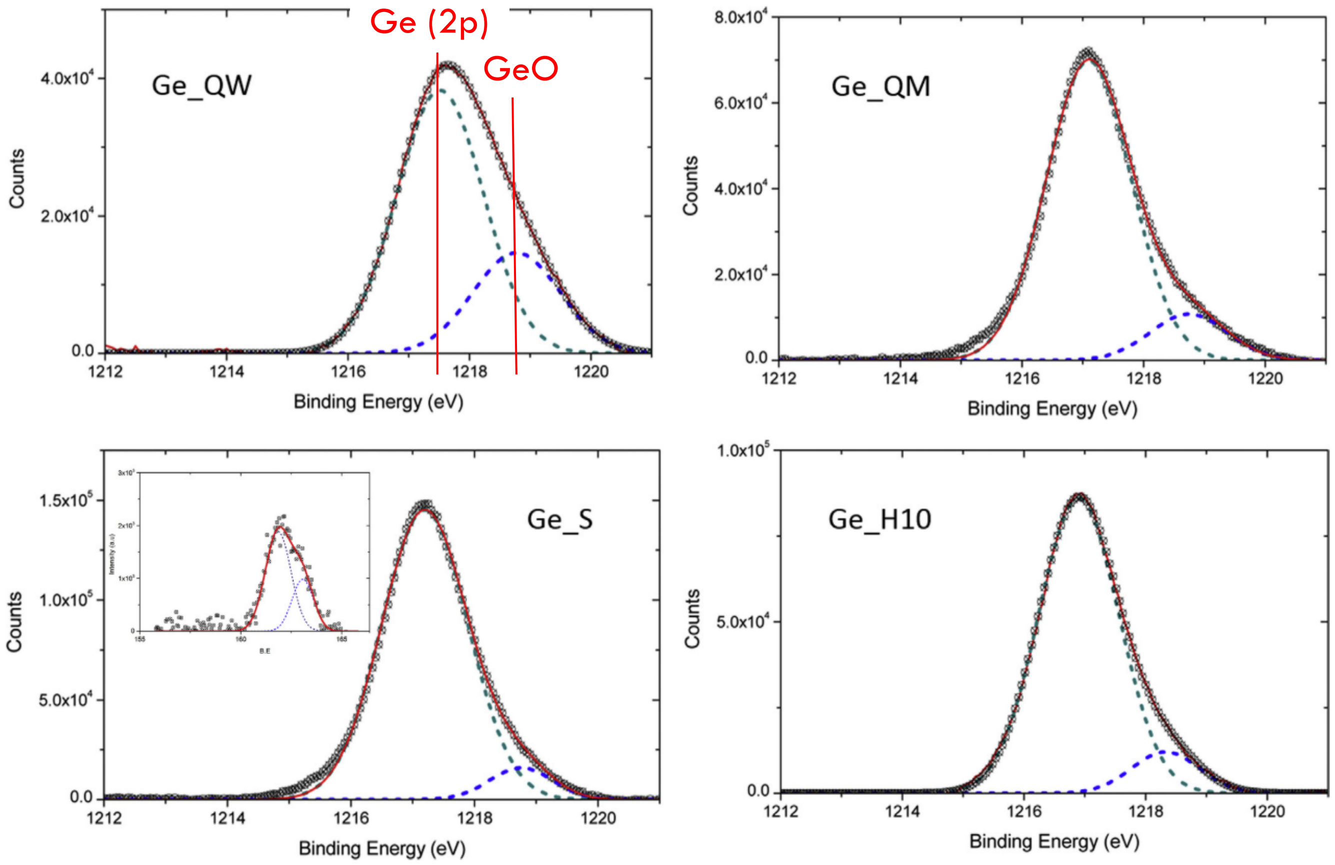
Passivation techniques: study the evolution of Ge – GeO – GeO_2

HNO_3/HF (3:1) etching and quenching bath:

- Water (Ge_W)
- Methanol (Ge_QM)
- Sulfide termination (Ge_S)
- Hydride termination (Ge_H10)

National Laboratories of Legnaro (LNL)
University of Padua (UNIPD)
& IKP Cologne

X-ray Photoelectron Spectroscopy (XPS)

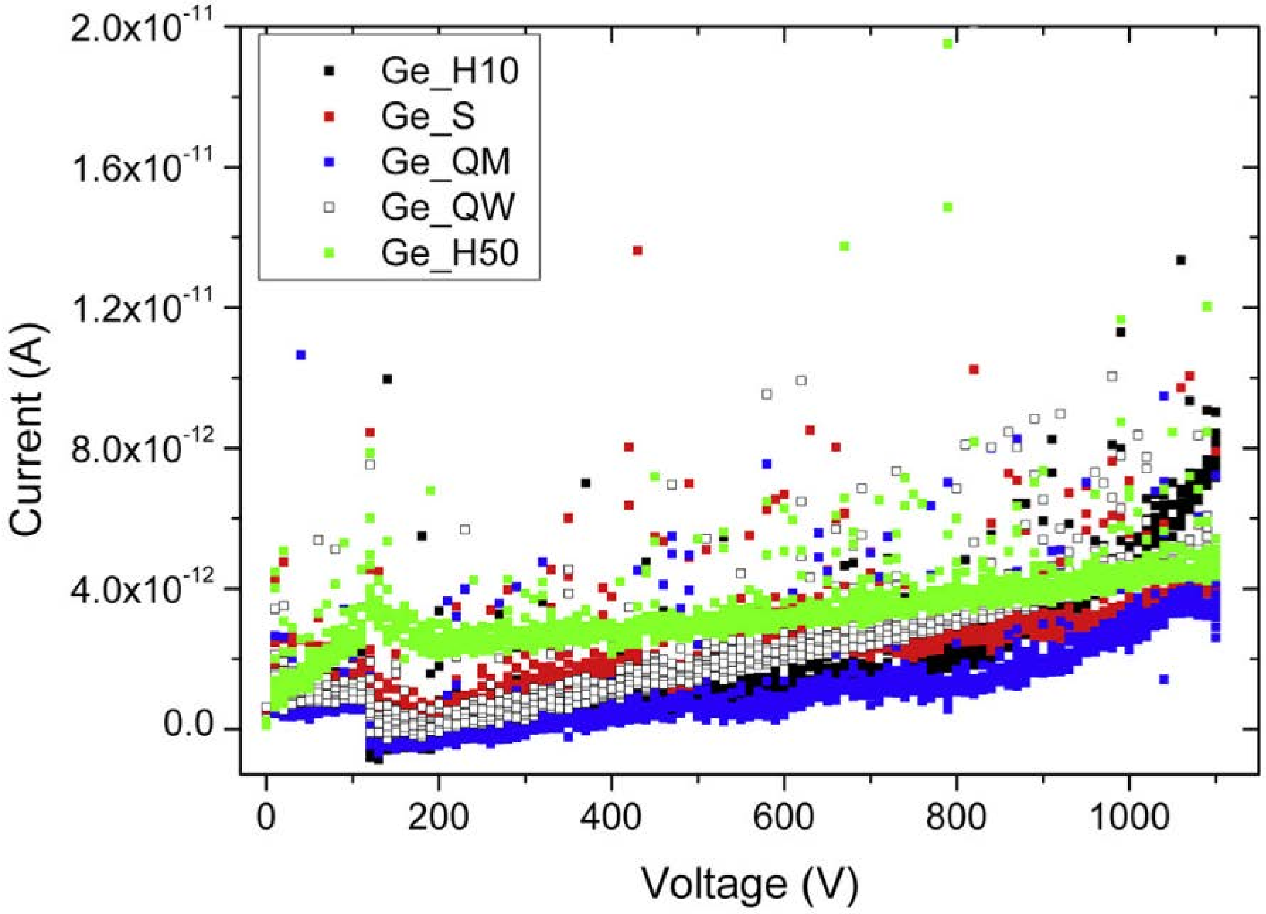


S. Carturan et al., **Mater. Chem. Phys.** 2015

HPGe gamma detector (Passivation test)

I-V Curve to determine the leakage current
(in diode configuration measurements at cryogenic temperature)

- Crystal is depleted (reverse bias)
- Crystal at 80 ÷ 90K



S. Carturan et al., **Mater. Chem. Phys.** 2015

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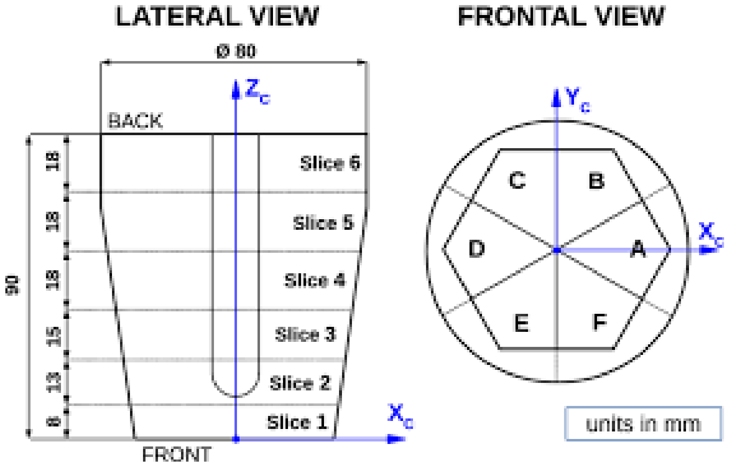
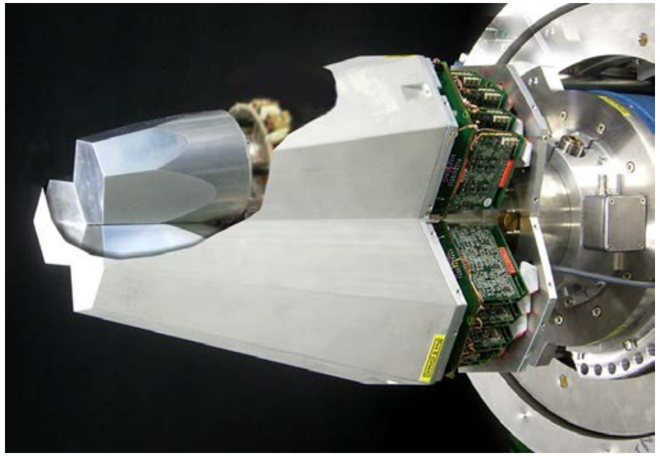
Gamma detector state of the art (AGATA)

Encapsuleted coaxial HPGe n-type detectors

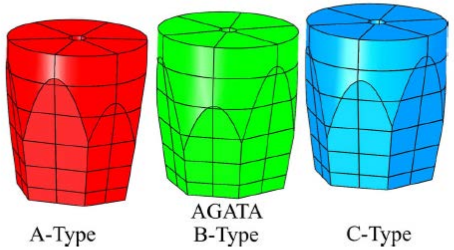
- 2Kg weight
- 80 mm diameter
- 90mm long
- 1 core inner contact
- 36 segments



ATC (AGATA triple cluster detector)



3 asymmetrical HPGe detector



J. Eberth et al. **Eur. Phys. J. A** (2023) 59: 179

Installation at LNL-INFN (13ATCs)

J.J Valiente al. **NIM** Phys. Res. **A** (2023) 1049

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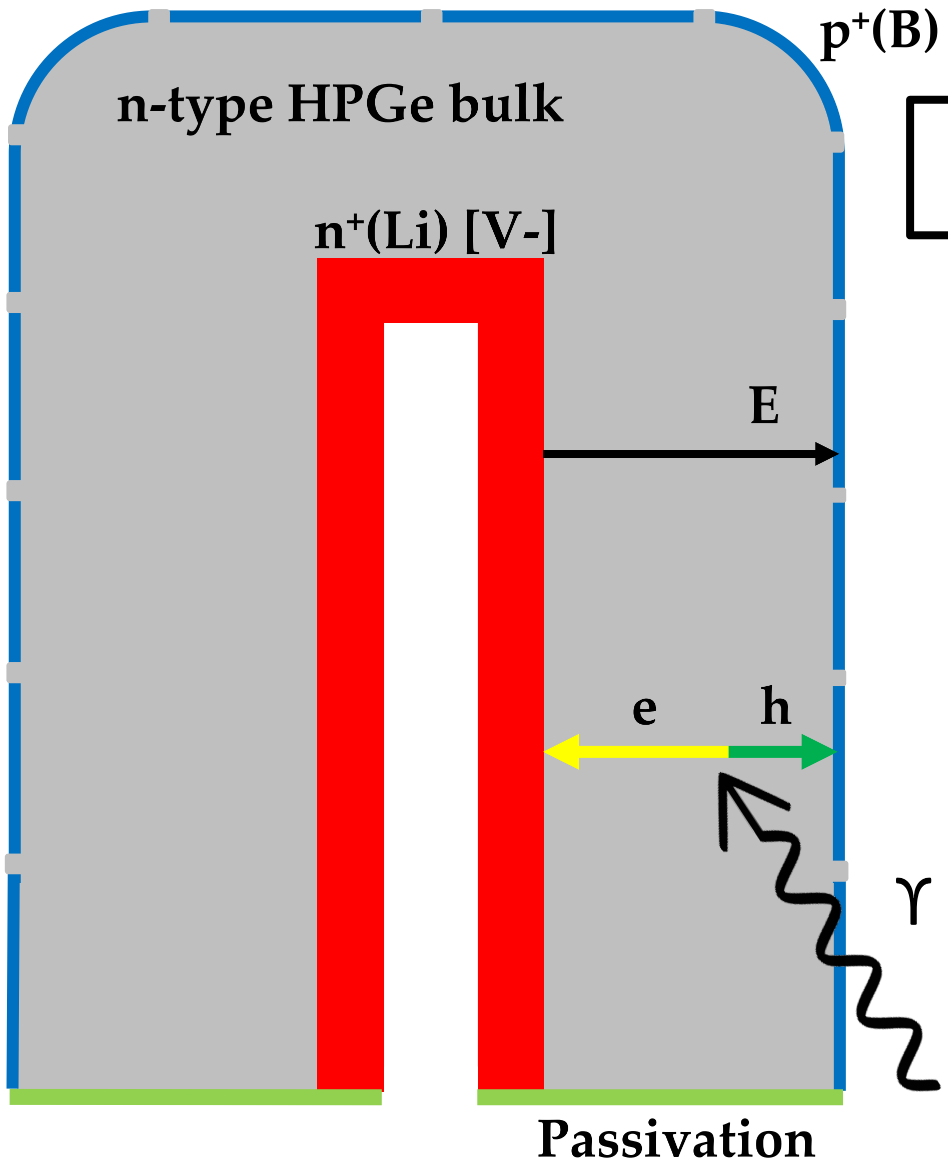
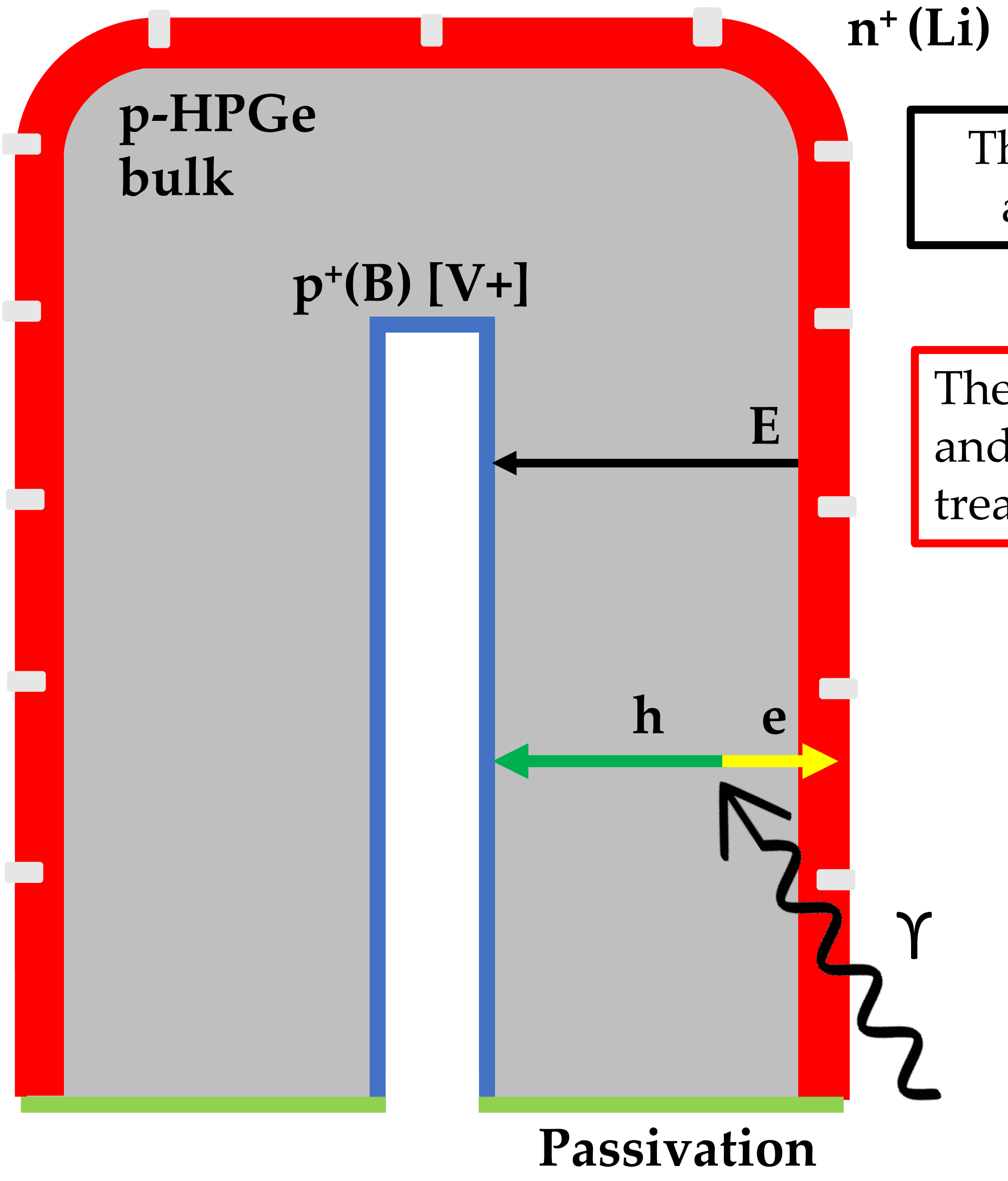
*Schematic coaxial segmented **n-type** detector*



The boron junction is thin, and easily segmentable

*Schematic coaxial segmented **p-type** detector*



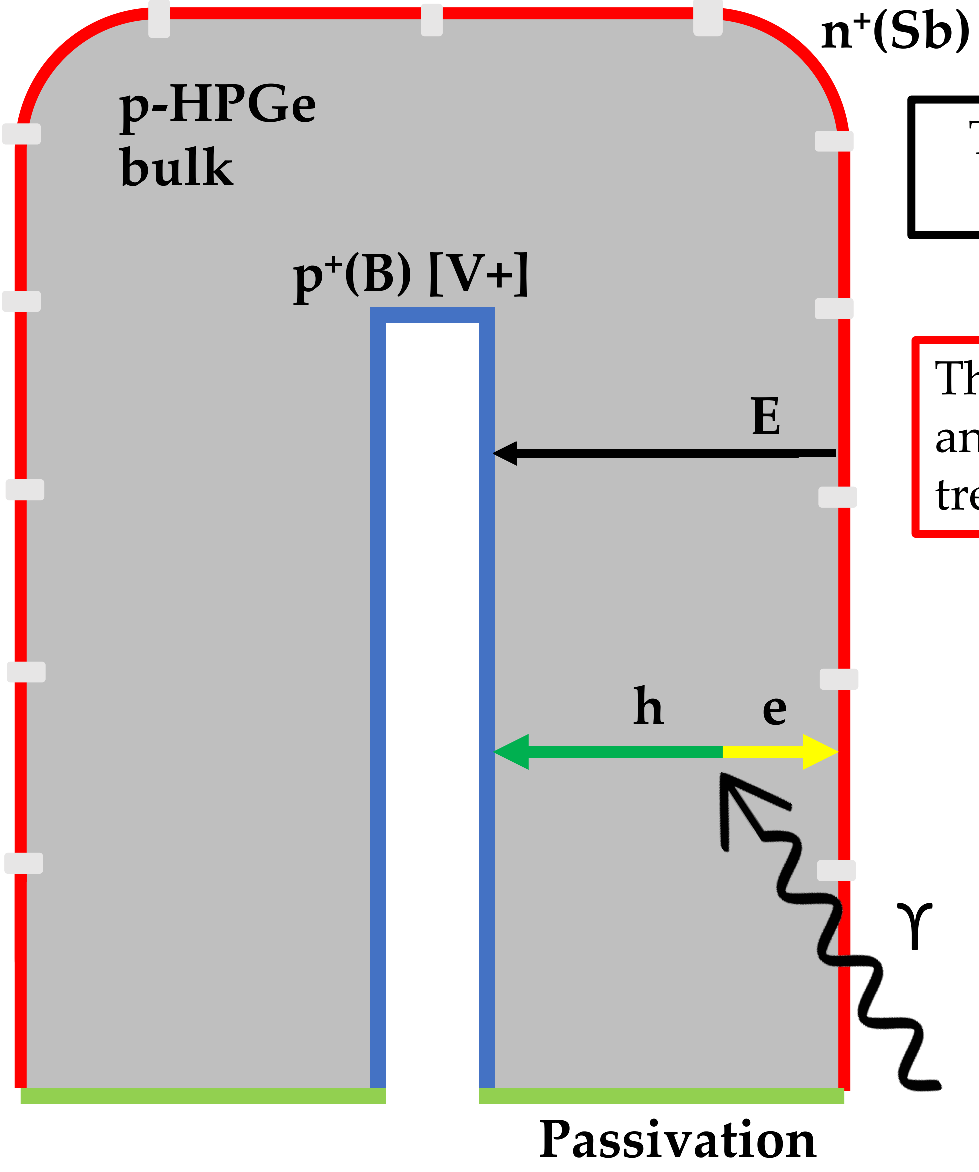
The boron junction is thin, and easily segmentable

The lithium junction is thick and not stable under annealing treatments

Polarity inversion demanded to test higher damage resistivity (hole trapping by neutron damage)

J. Eberth et al. **Eur. Phys. J. A** (2023) 59: 179

*Schematic coaxial segmented **p-type** detector*



The boron junction is thin, and easily segmentable

Polarity inversion demanded to test higher damage resistivity (hole trapping by neutron damage)

The lithium junction is thick and not stable under annealing treatments

J. Eberth et al. **Eur. Phys. J. A** (2023) 59: 179

Thin and thermally stable n-type dopants (**Pulsed Laser Melting**).

p-HPGe with segmentable n+ junction collecting electrons.

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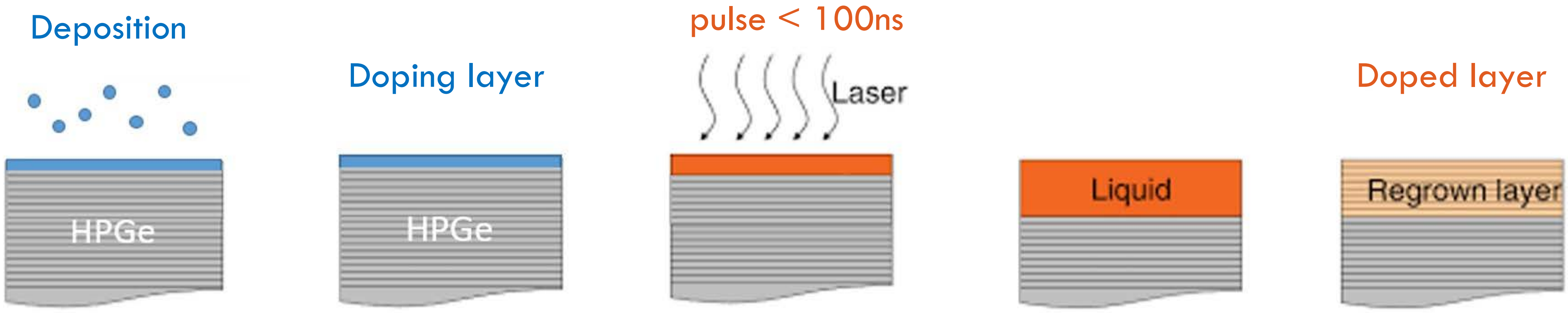
New contact/junction on HPGe: PLM (Pulse Laser Melting)



Advantages:

- Melting temperature is reached - short time (<100 ns)
- Only the surface (< 200 nm) is melted, the bulk is at room temperature
- High dopant concentrations with very sharp dopant profile
- Doping with heavy elements without crystal damage
- Very clean process suitable for preserving the Ge hyperpurity
- Suitable for complex contact geometries (**segmentation**)

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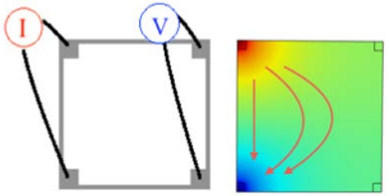
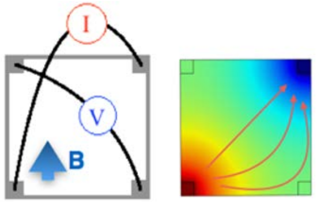
New contact/junction on HPGe: PLM on HPGe crystal

Van Der Pauw



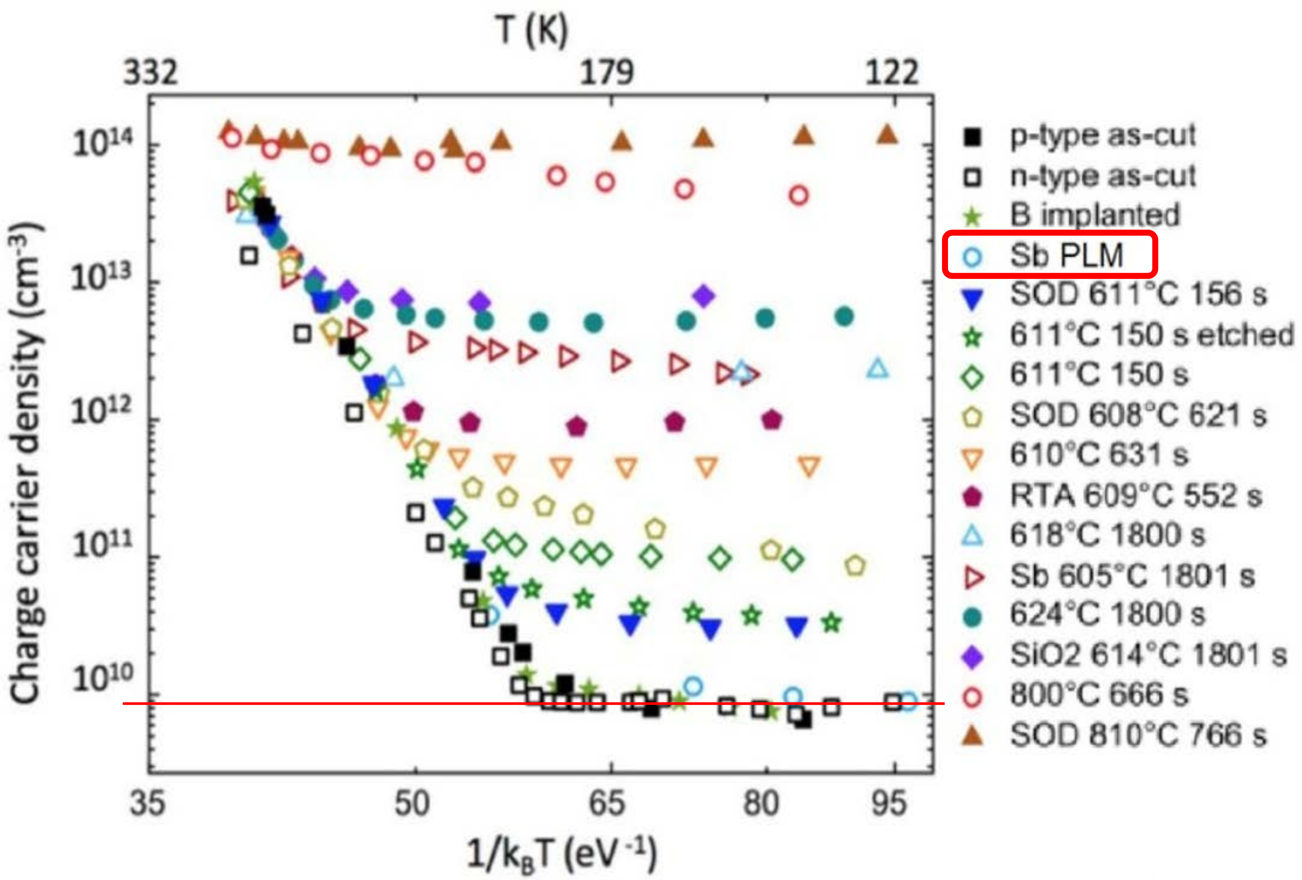
Sheet resistance

Hall



Charge carriers

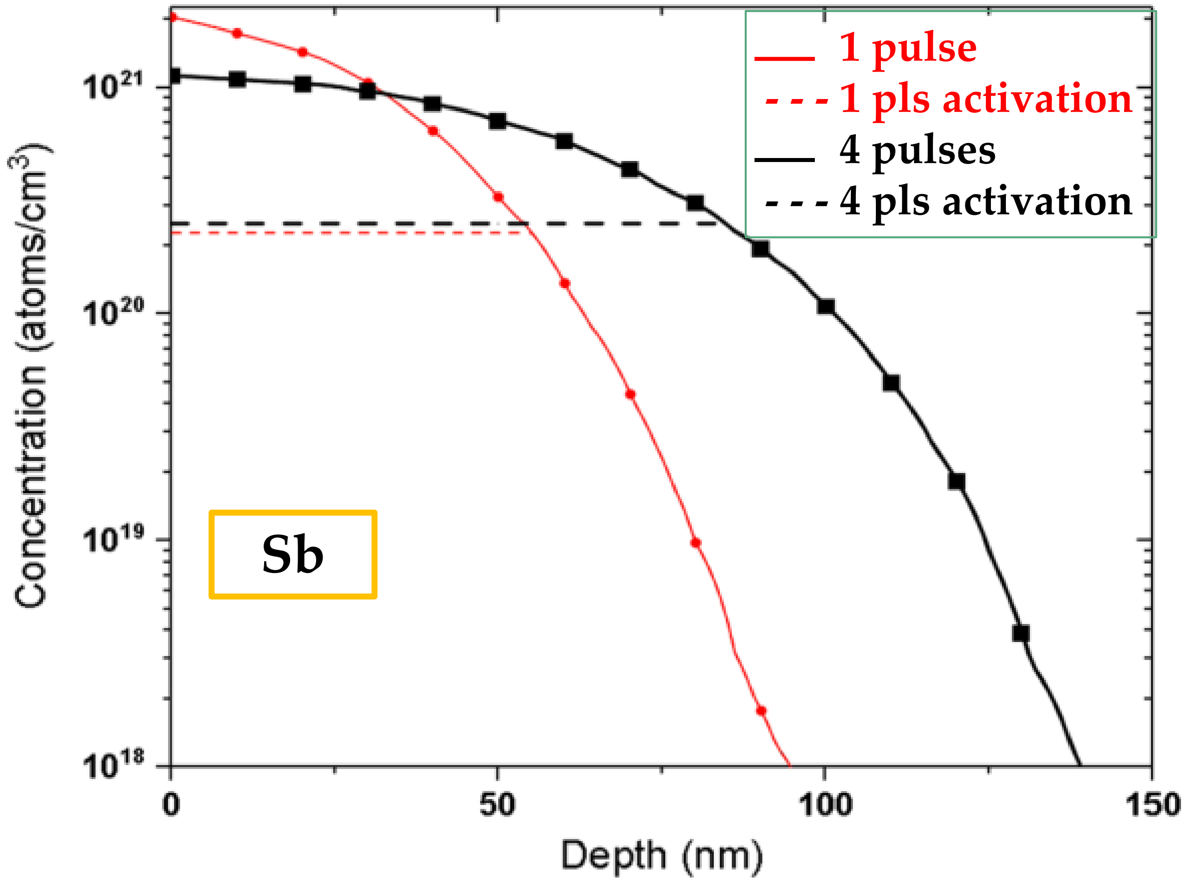
Impurities concentration in Ge bulk

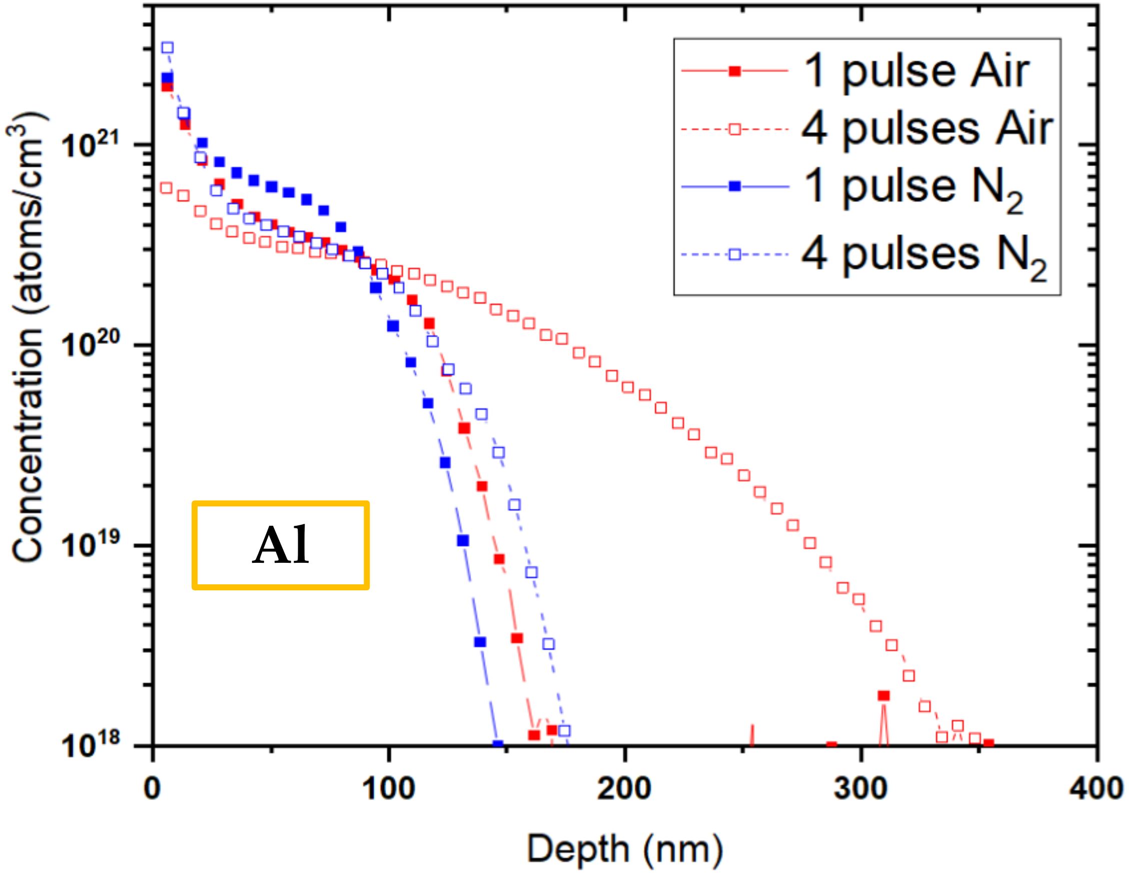


V. Boldrini et al., **Journal of Physics D: Applied Physics** (2018) volume 52, 3

New contact/junction on HPGe: Chemical concentration profile

SIMS (Secondary Ions Mass Spectrometry)





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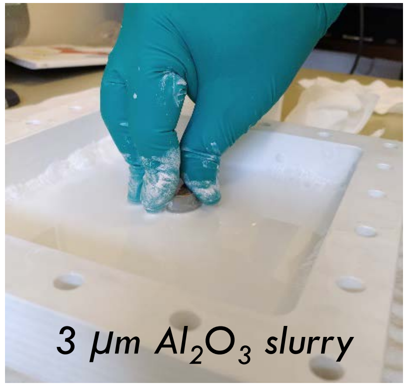
New contact/junction on HPGe: Surface preparation

Grinded surface
(opaque)

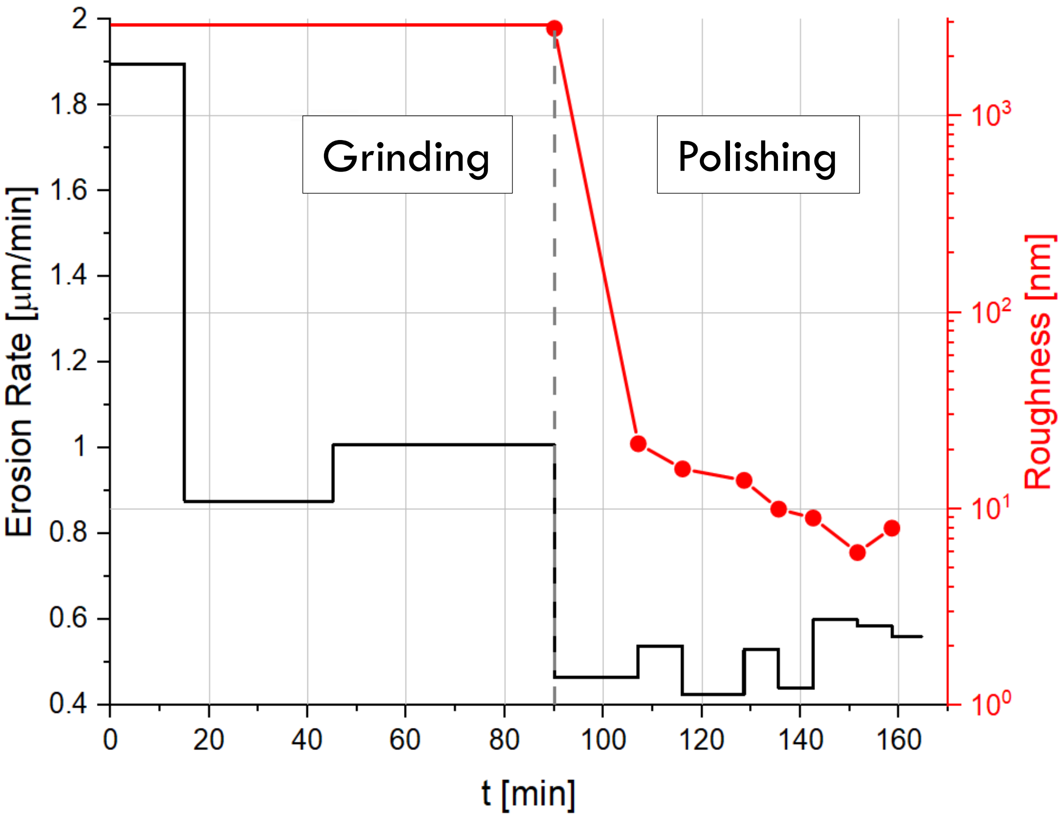


Etched surface
(High waviness,
low roughness)

(3:1) HNO_3 : HF

Polished surface
(Low waviness,
low roughness)

rotating disc rinsed
with H_2O_2 1%
(pH 12 with KOH)



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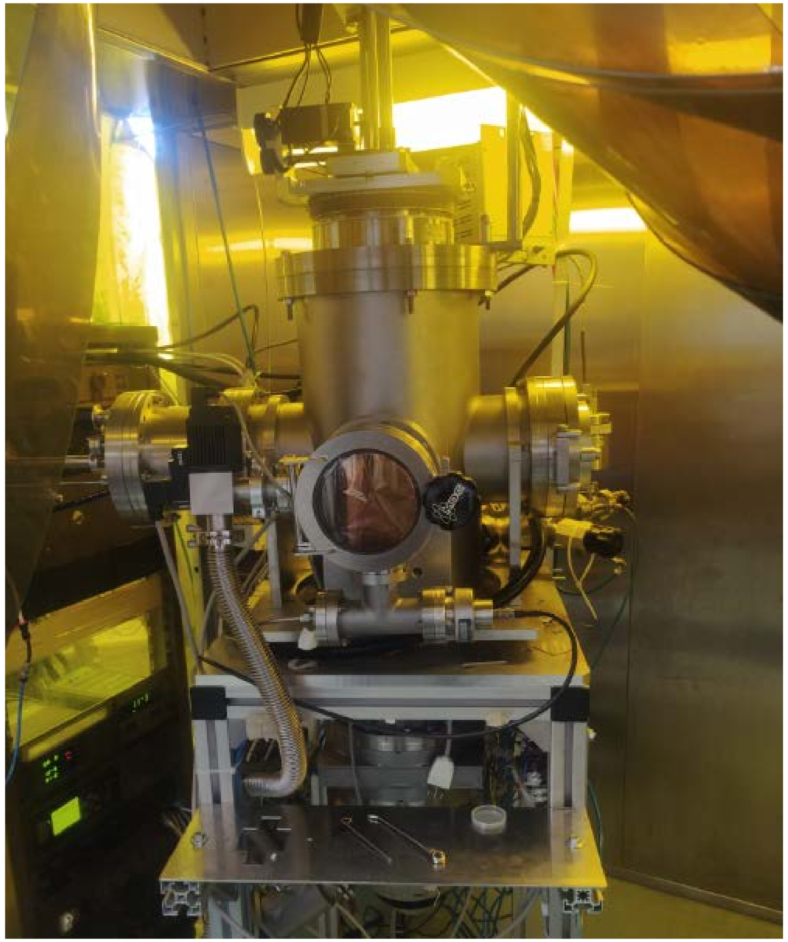
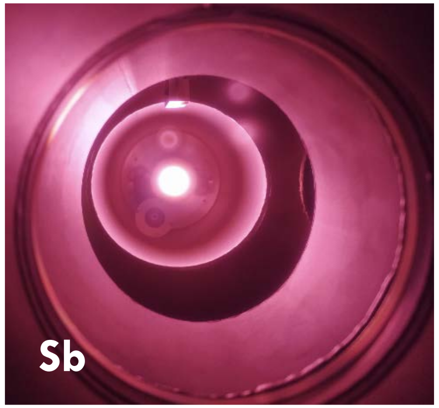
New contact/junction on HPGe: PVD Sputtering deposition

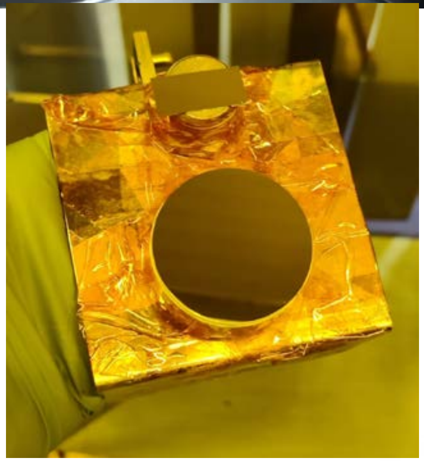
Magnetron Sputtering deposition



Sb material



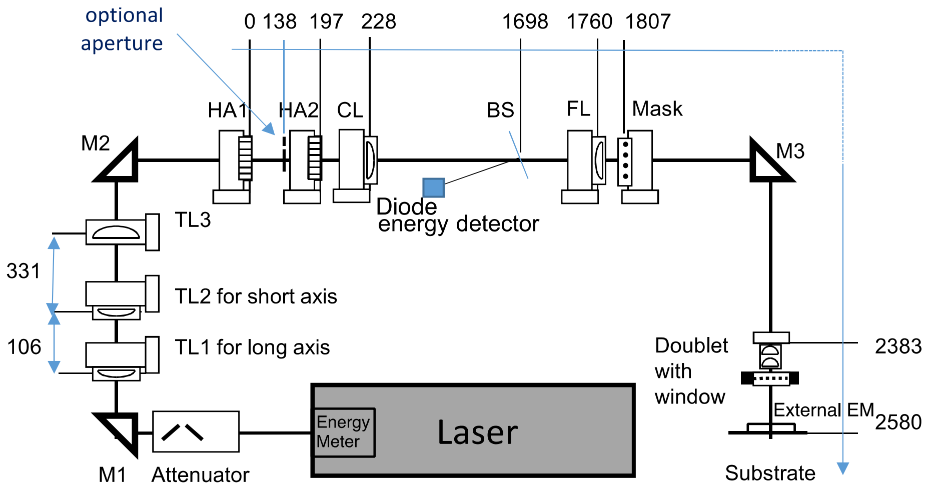
Al / Ge material

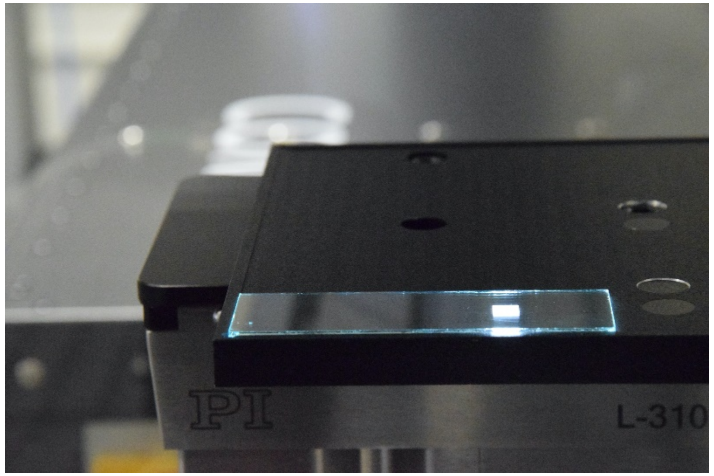


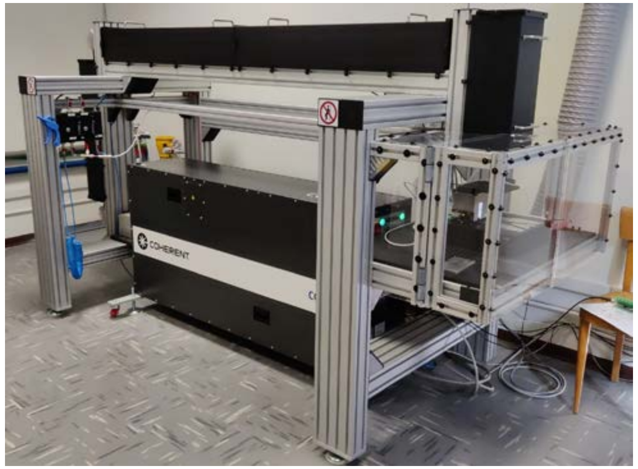
n^+	Sb	2 nm
p^+	Al (Ge cap)	4 nm (10 nm)

New contact/junction on HPGe: PLM Laser tecnology

Excimer KrF laser







- λ=248 nm, 22 ns
- Frequency: 1-10 Hz
- ED= 50-1300 mJ/cm^2
- Square 5x5mm^2 spot
- Homogeneity: < 2%
- lateral resolution <30 µm
- Motorized XYZ stage

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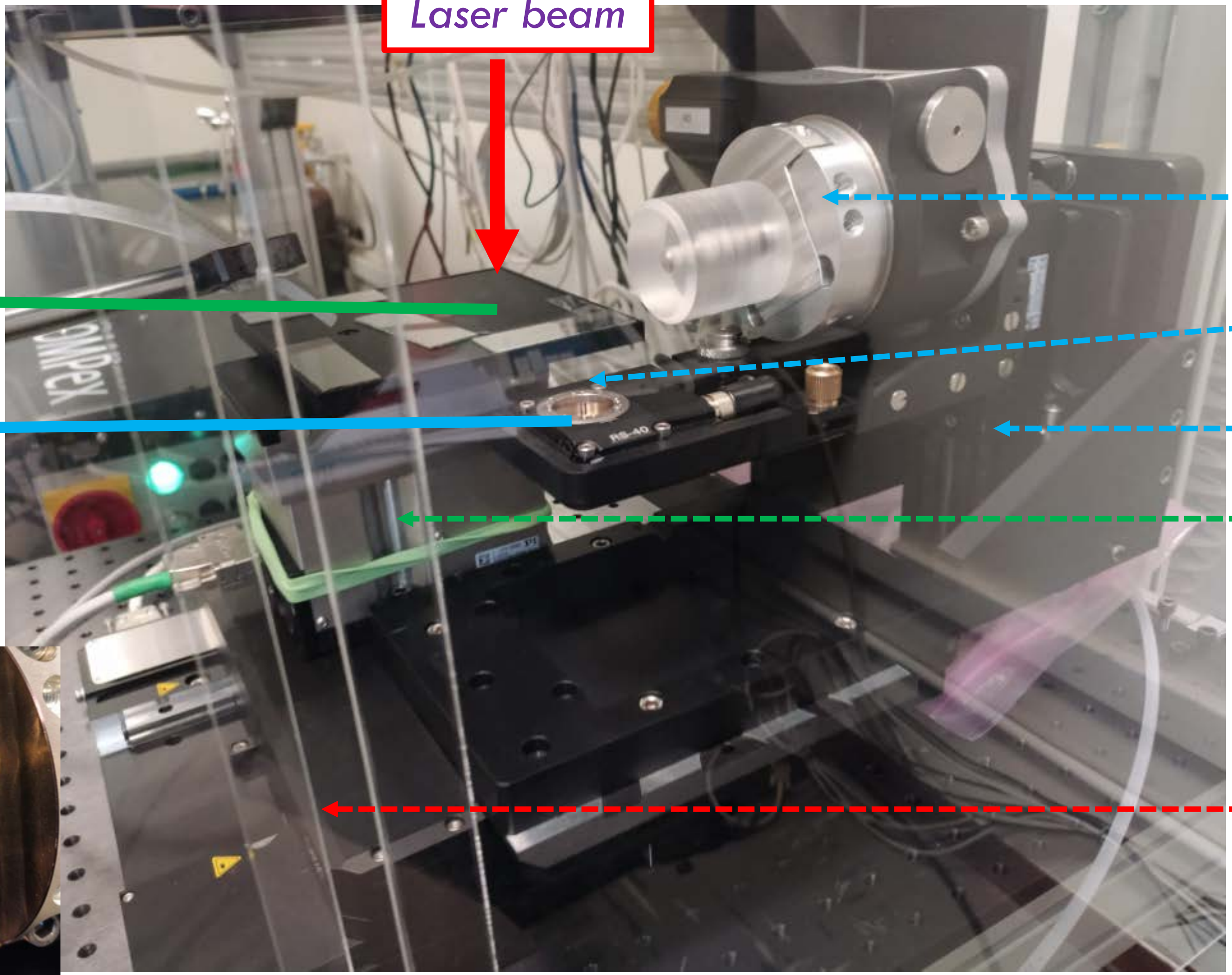
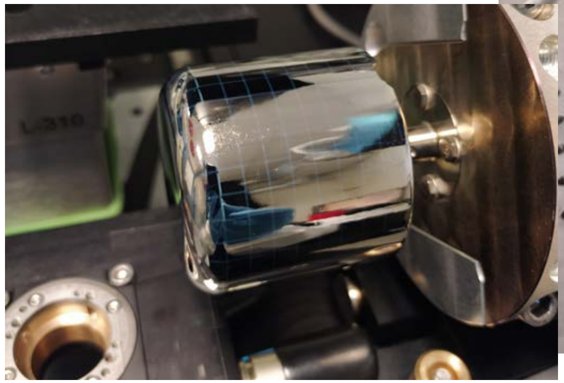
New contact/juntion on HPGe: PLM Laser tecnology





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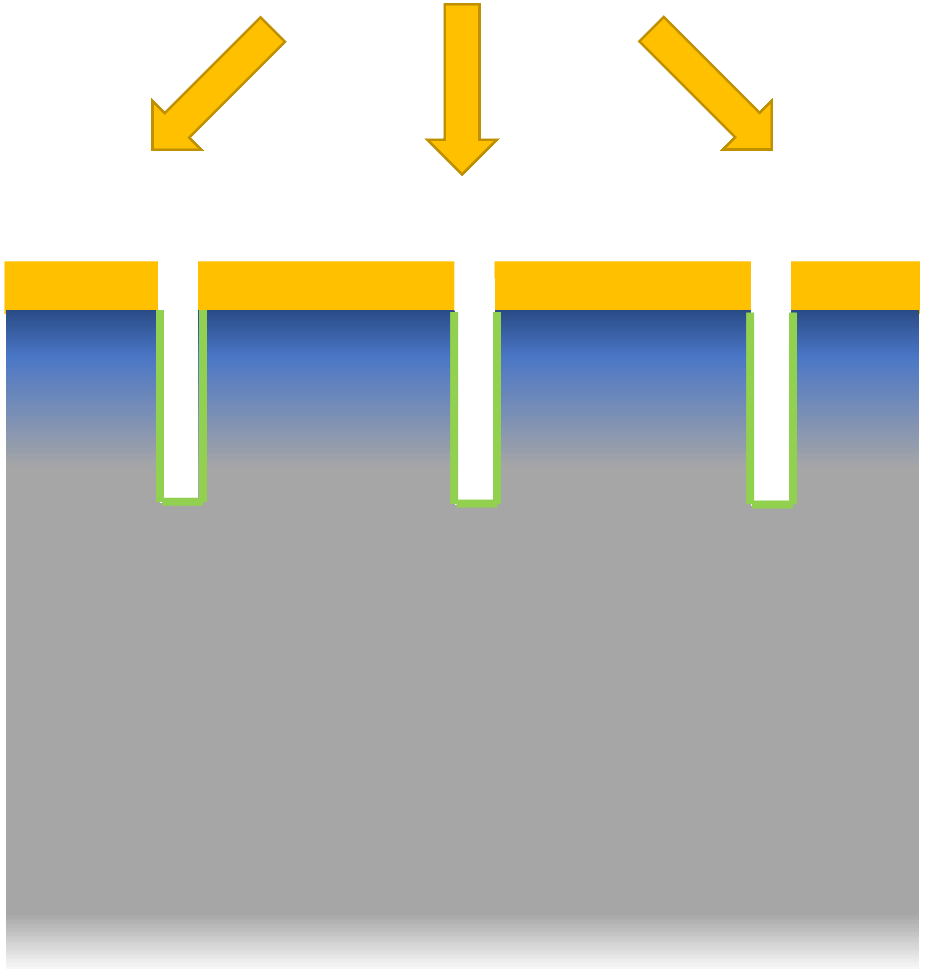
PLM contact/junction: 1° type Segmentation

Au target

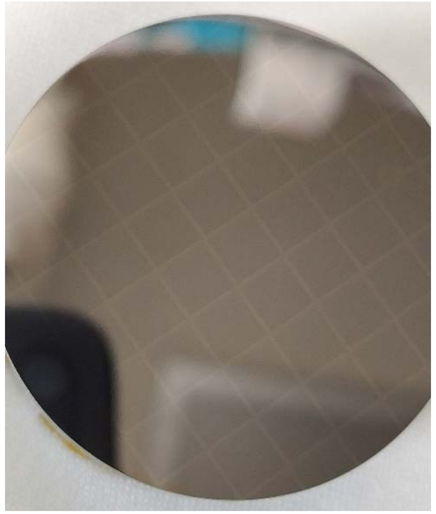


Full area ⇐ PLM

Au deposition
100 nm PVD deposition of Au in Ar plasma with ultrapure target in vacuum (10^{-6} mbar)

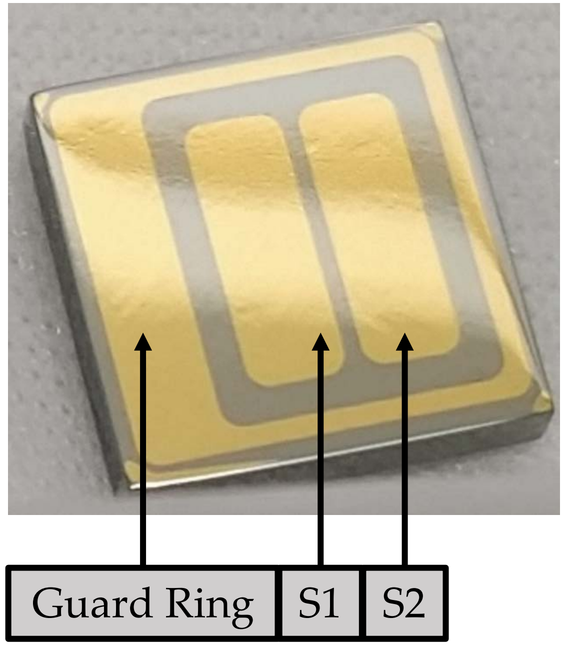
Photolithography
Photoresist deposition, baking, exposure and development, followed by Au stripping and resist removal.

Intercontact gaps passivation
(3:1) HNO_3 : HF etching followed by chemical quenching passivation.



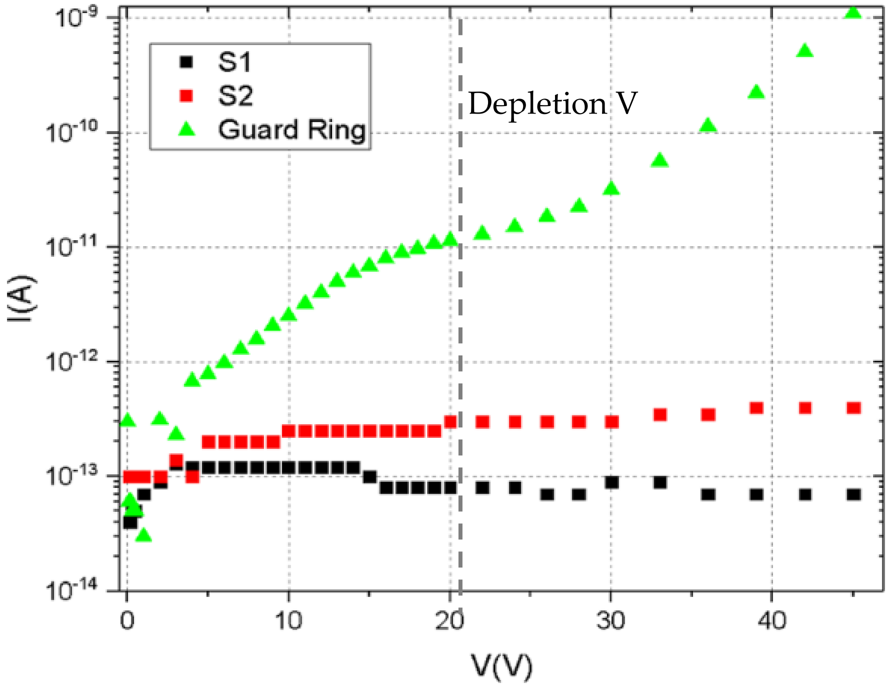
Thin planar HPGe detectors

Sb n$^+$ junction, p-type HPGe, B p$^+$ L=10mm, t=2mm



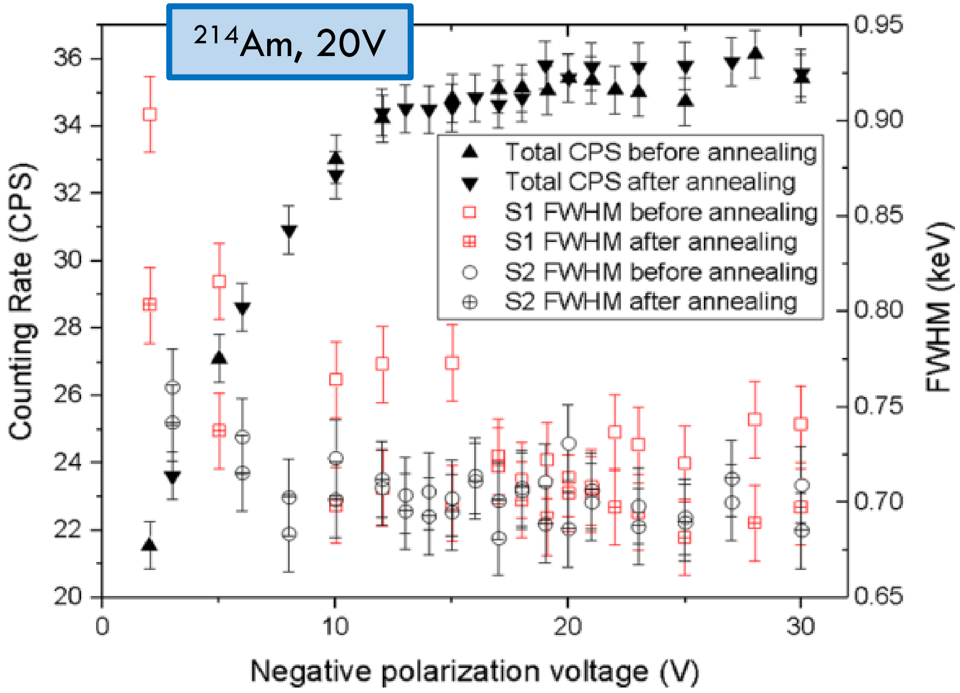
Measured before and after recovery annealing and re-passivation to test junction stability

Electrical test: reverse I-V characteristics



Gamma ray test: 241Am spectra acquisition

Annealing at 105°C



S. Bertoldo et al., **Eur. Phys. J. A** (2021) 57:177

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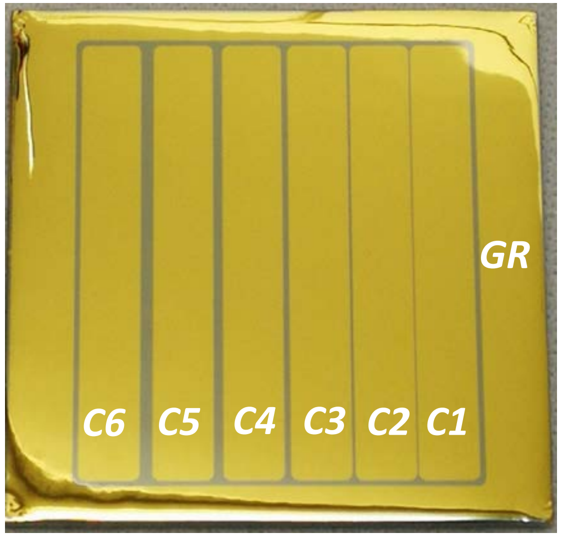
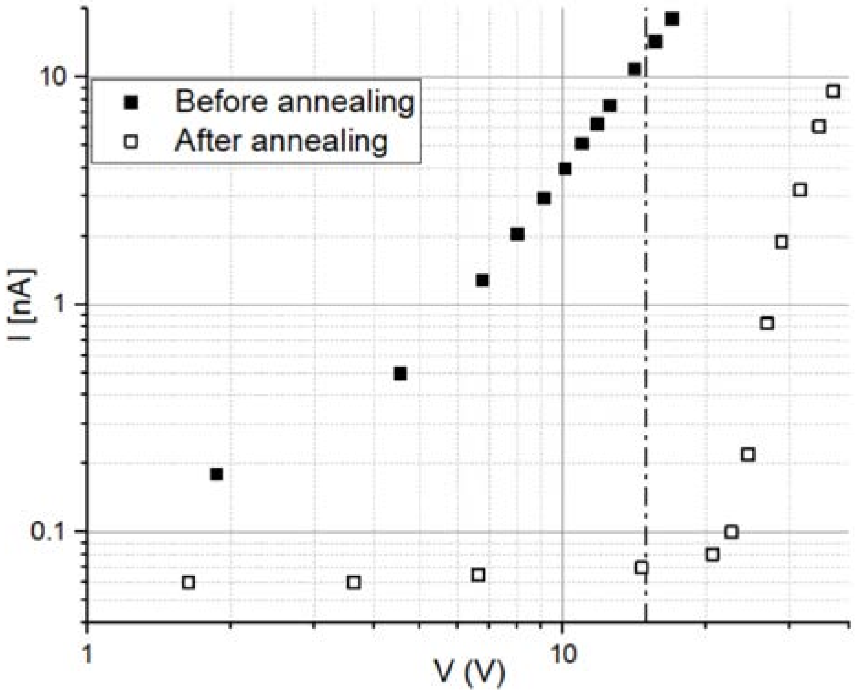
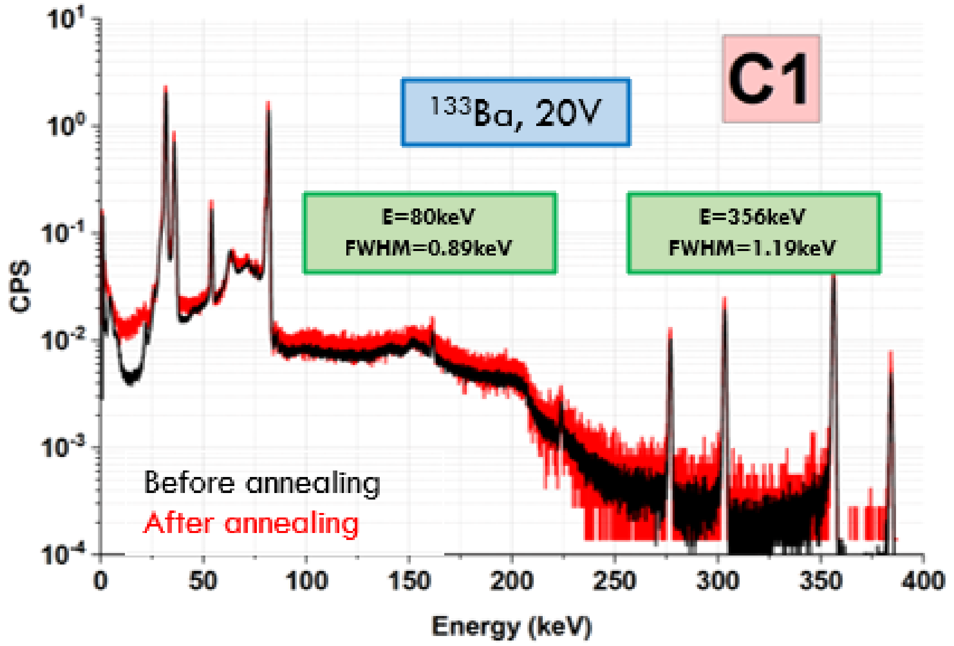
Thin planar HPGe detectors

Sb/p-HPGE/Al, L=35mm, t=2mm



Minimum gap tested 0.1mm





W. Raniero et al., **Il NUOVO CIMENTO 44 C** (2021) 154

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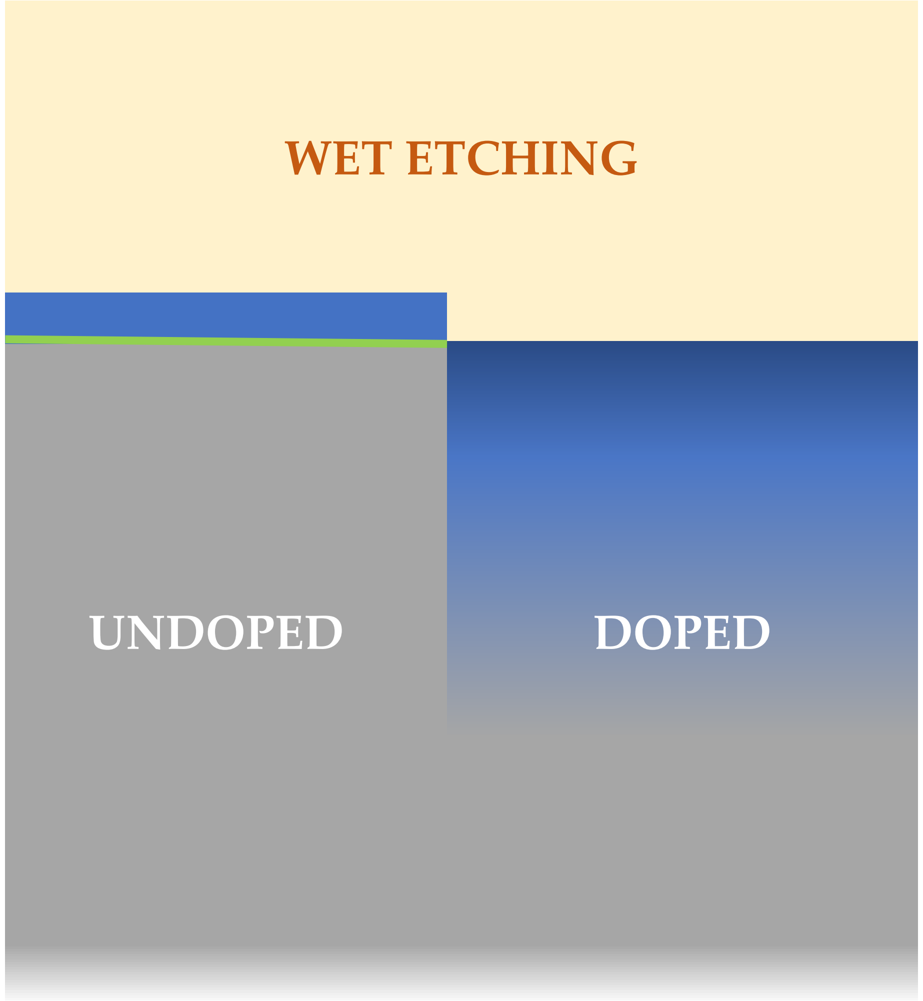
PLM contact/junction: 2° type Segmentation

Full area ⟸ **PLM** ⟹ Partial area



Lithographic process using selective etching solutions:

- Hot pure H_2SO_4 for Sb deposition (preserve Sb junction)
- H_2O_2 for GeP deposition (slowly etches everywhere)
- Kern solutions (H_2O_2, H_3PO_4, Ethanol) for Al-Ge deposition (preserve Al junction)

exposure and development, followed by Au stripping and resist removal.

Intercontact gaps passivation

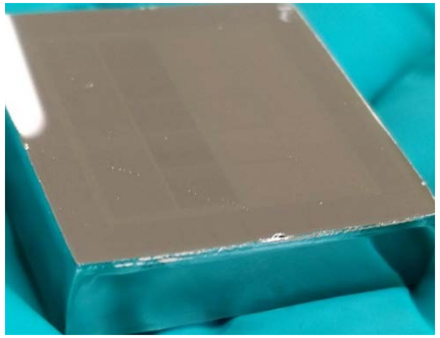
(3:1) HNO_3 : HF etching followed by chemical quenching passivation.

Selective etching

Removal of untreated dopant using selective etchants to protect the near junction.

Chemical passivation

Passivation of undoped surfaces with suitable solutions.

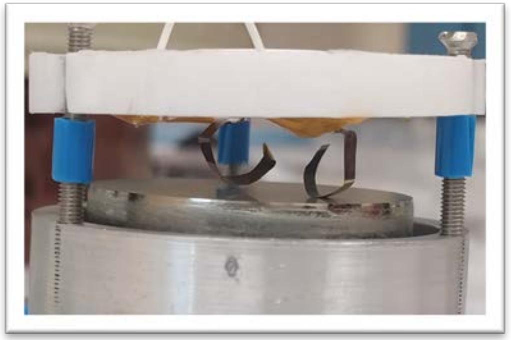


Thick planar HPGe detectors

Sb/p-HPGE/Al,
L=35mm, t=10mm

n+ junction
(spring contact)

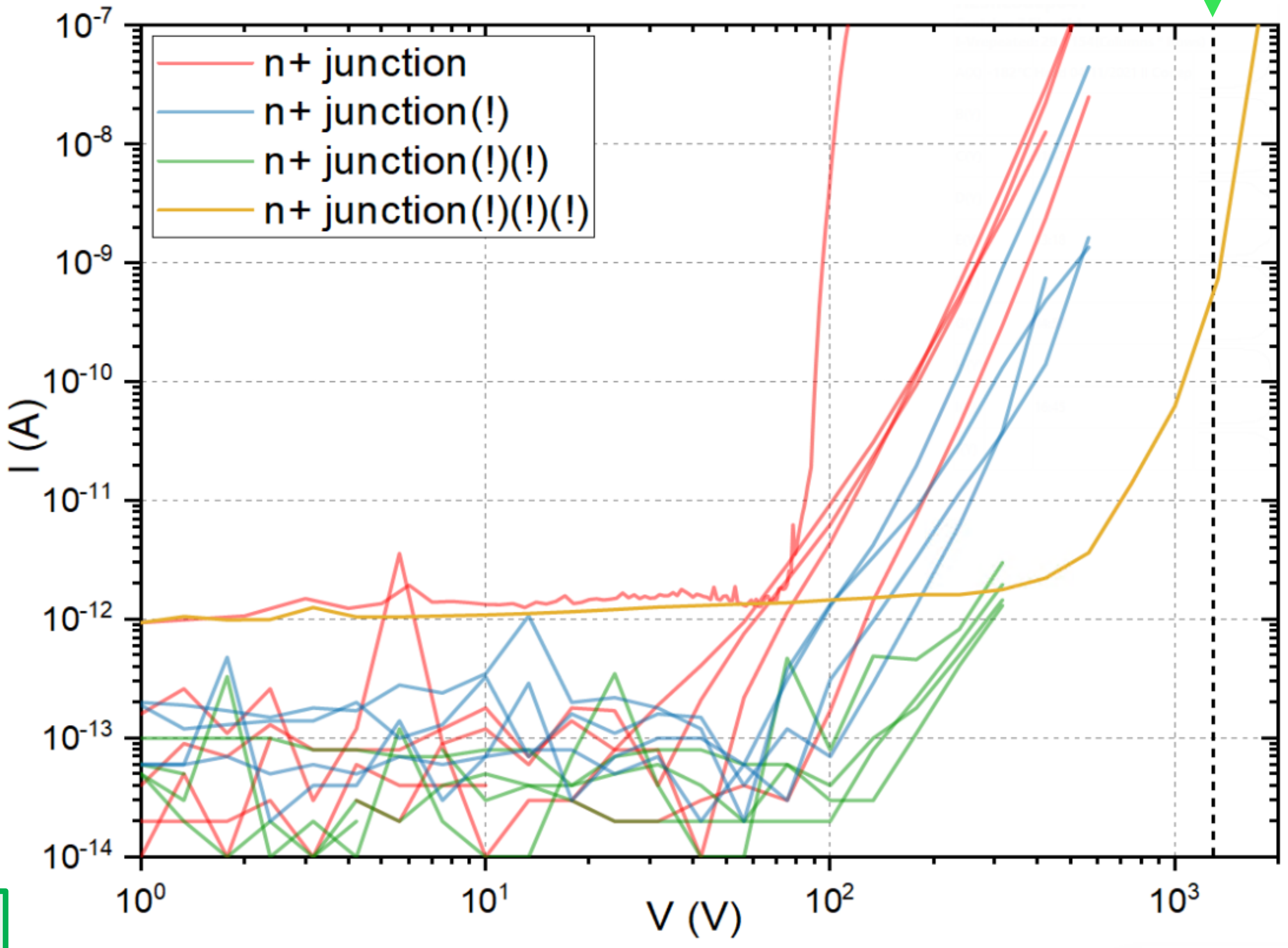
n+ junction (!) / (!)(!)
indium pad



n+ junction (!)(!)(!)
elastic tabs

Sb/p-HPGE/Al,
D=40mm, t=20mm

$V_{depl.}$: 1300V



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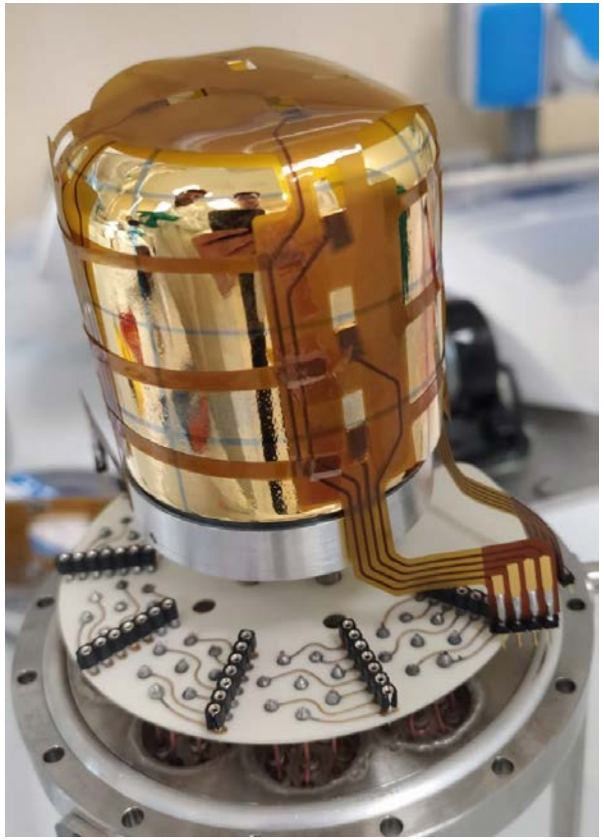
Coaxial HPGe detectors: 3D Photolitography Segmentation

Robot 3D photolitography

D=50mm, H=50mm

Flexible PCB contacts



Coaxial Photolithography: Robot 3D

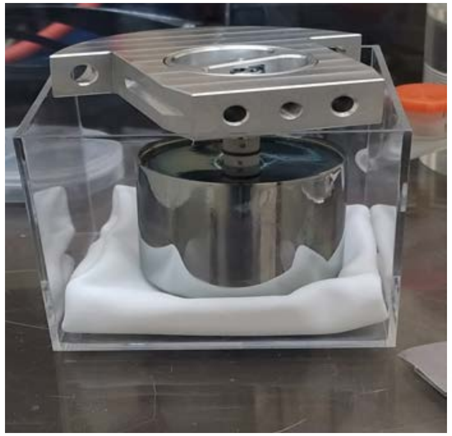
The laser micrometer measures the surface after a rotation of the coaxial detector while keeping the robot in the same position

Coaxial Dummy

Misalignment of the segmentation lines at the top of the sample

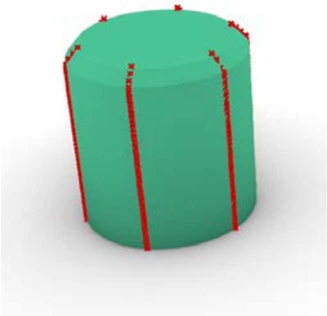
The error is non-reproducible and is caused by the gripping system of the coaxial detector and the hole in the crystal itself

3D mapping of the coaxial detector and obtaining its coordinates relative to the robot's coordinate system with an accuracy of less than 0.1mm



Coaxial Photolithography: Robot 3D

1


2


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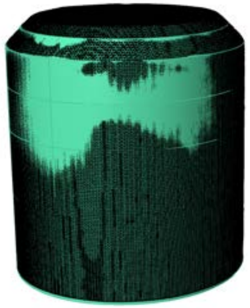

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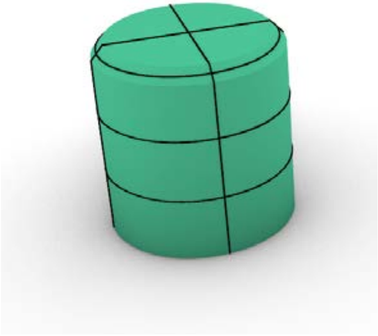

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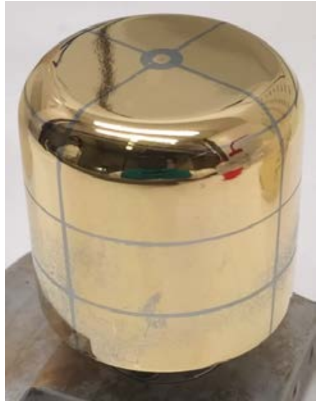


1. Mapping of the cylinder through vertical lines: line formed by a series of points
   Each point is determined by the robot's position + laser micrometer measures

2. 3D reconstruction via lofting technique.

3. Comparison with a professional 3D scanner , Accuracy <0.1mm.

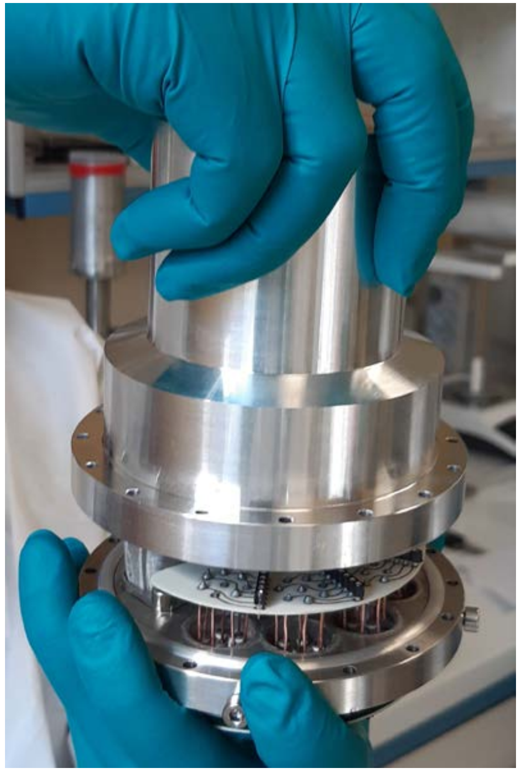
4. Construction of the pattern to be lithographed in the robot's coordinate system

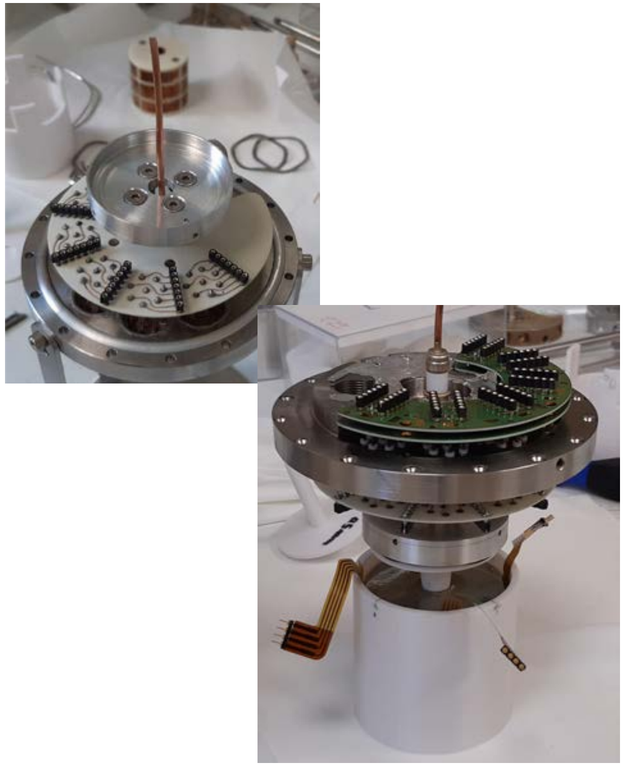
5. Photolithography carried out by the robot

UV photolithography robot

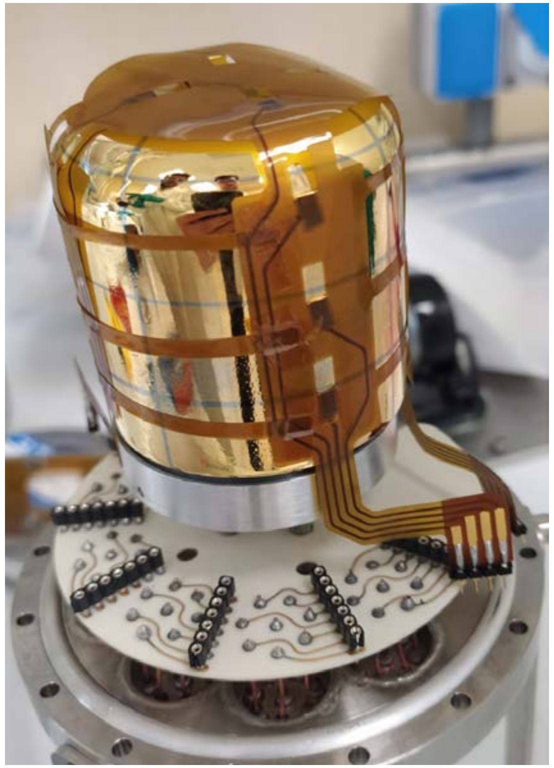
Encapsulation of Coaxial detector

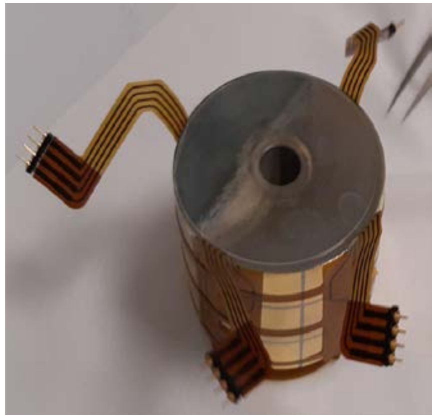
Vacuum - tight canister





Flexible Kapton PCB





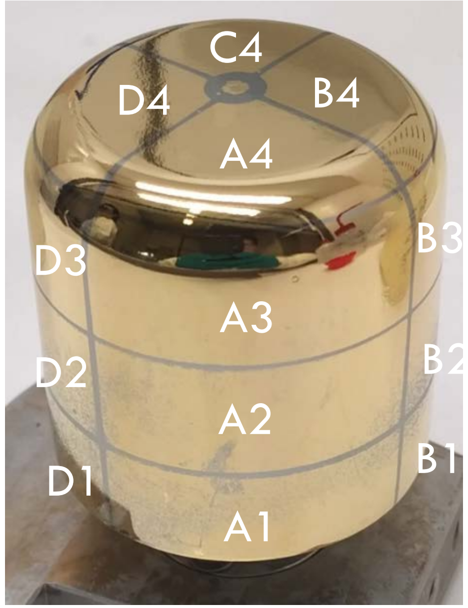
S. Capra et al, **JINST 19 C01011** (2024)

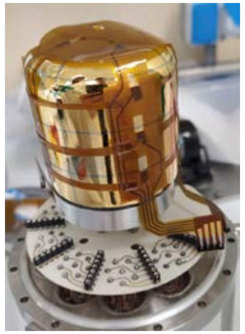
Segmentation Test of Coaxial detector

T= 25°C

Ω	A1		B1		C1		D1
A2	**17.8**	B2	**16.6**	C2	**16.4**	D2	**22.1**
A3	**23.6**	B3	**21.3**	C3	**21.5**	D3	**27.3**
A4	**26.8**	B4	**23.8**	C4	**23.6**	D4	**30.5**

Ω	A1		A2		A3		A4
B1	**22.5**	B2	**18.0**	B3	**17.7**	B4	**18.0**
C1	**27.0**	C2	**19.8**	C3	**19.4**	C4	**20.1**
D1	**21.7**	D2	**16.3**	D3	**18.1**	D4	**17.4**





T= 80°K

GΩ	C1	C3	D2	D4
Up	**0.4**	**20**	**1.4**	/
Down	/	**0.6**	**7.5**	**3.0**
Right	**6.0**	**7.7**	**5.3**	**31.3**
Left	**4.3**	**25.0**	**0.1**	**12.8**

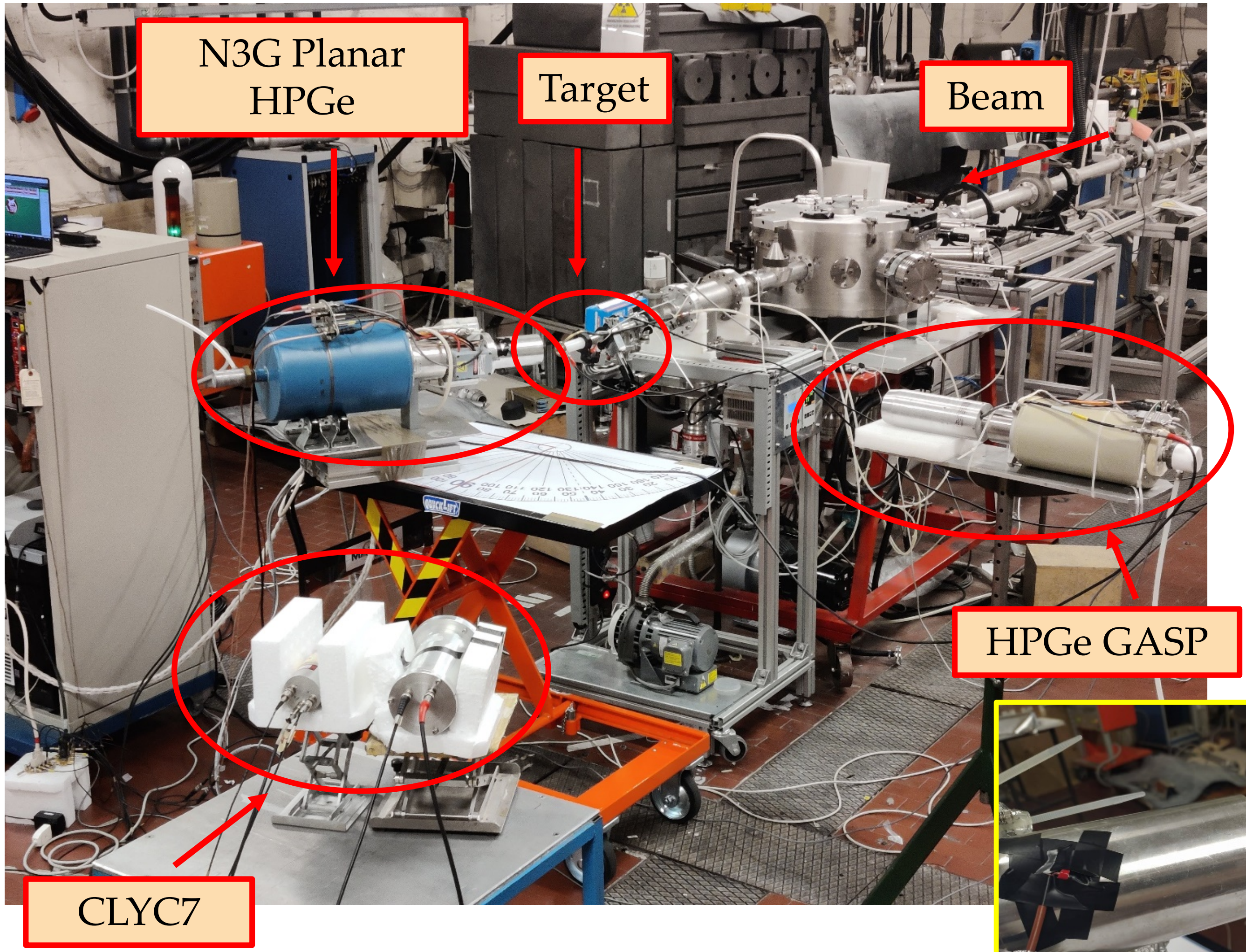
GΩ	A1	B2	B4
Up	**8.9**	**21.7**	/
Down	/	**3.1**	**27.8**
Right	**62.5**	**0.2**	**5.0**
Left	**62.5**	**11.4**	**12.5**

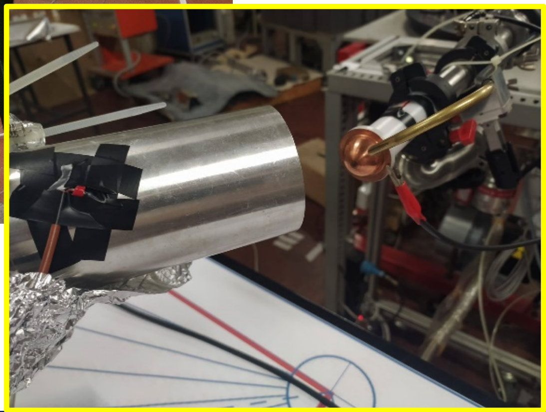
High resistance between the segments is measured, exceeding 100 MΩ

OUTLINE

- Introduction: HPGe gamma detectors
- Gamma detector state of the art
- *PLM (Pulse Laser Melting):* Next generation of segmented contact/junction on HPGe detectors
- PLM Planar gamma segmented detectors
- PLM Coxial gamma segmented detectors
- Neutron damage in PLM planar segmented detectors

Neutrons damage on planar PLM segmented detector





380nA 4MeV proton beam
^{7}Li target, 100µm

Reaction: $^{7}Li\ (p,n)\ ^{7}Be$

Prototype detector is located at 30° 9.5 cm

Neutrons are directly measured with

- CLYC7 scintillators, 30° 2 m
- GASP HPGe γ detector, 90° 1 m

$^{7}Be + e^- \xrightarrow{53.3\ days} {}^{7}Li$ ⟿ 477.6 keV

R. Escudeiro at all. "**Neutron radiation damage on a planar segmented germanium detector**" proceeding Presented at the XXXVII Mazurian Lakes Conference on Physics, Piaski, Poland, September 3-9, 2023

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Neutrons damage on planar PLM segmented detector: process steps

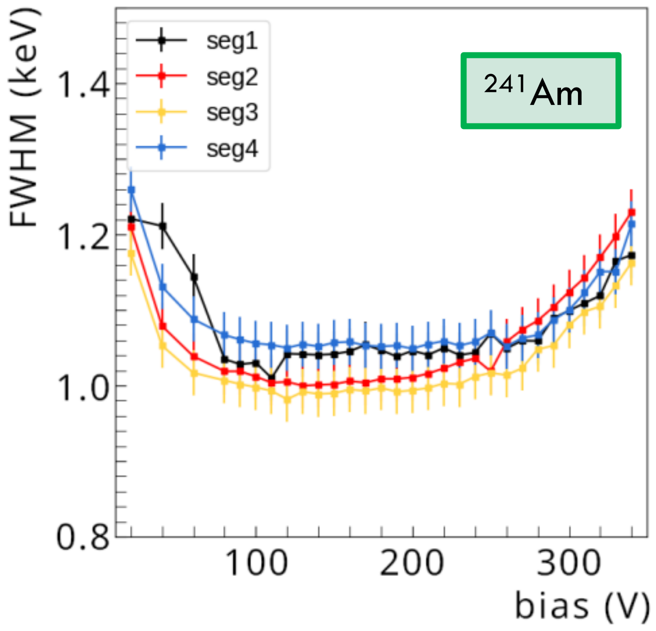
Detector prototype:
Sb/p-HPGE/Al,
L= 32mm,
t= 8.6mm
4 contacts + guard ring

1. The performance test of the new detector
2. The performance test of the detector after 1st run of neutron damage
3. The performance test of the detector after the annealing recovery
4. The performance test of the detector after 2st run on neutron damage

Neutrons damage on planar PLM segmented detector: before run

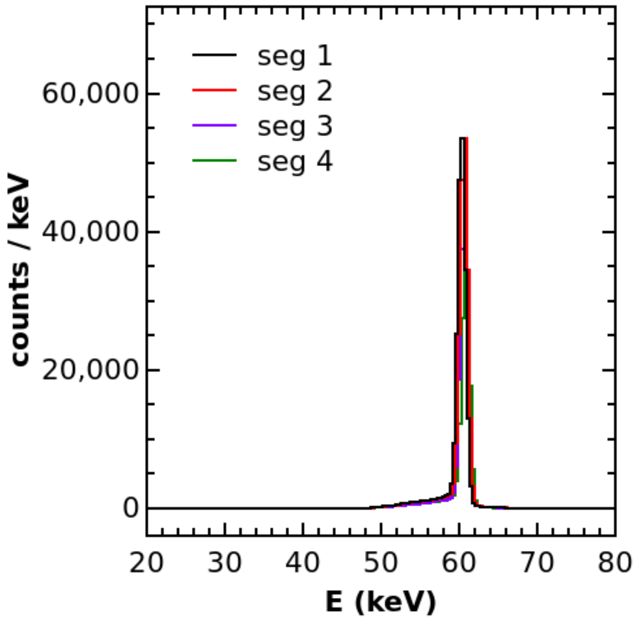
Detector prototype:
Sb/p-HPGE/Al, L= 32mm, t= 8.6mm
4 contacts + guard ring

Energy resolution in the lab

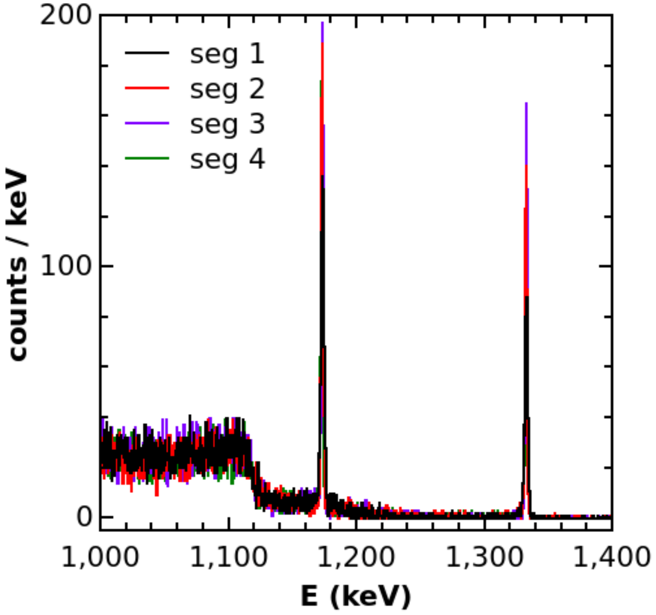


Starting resolution at **80V** operational bias before the neutron damage run

^{241}Am
E = 59,5 keV
FWHM < 1,1 keV



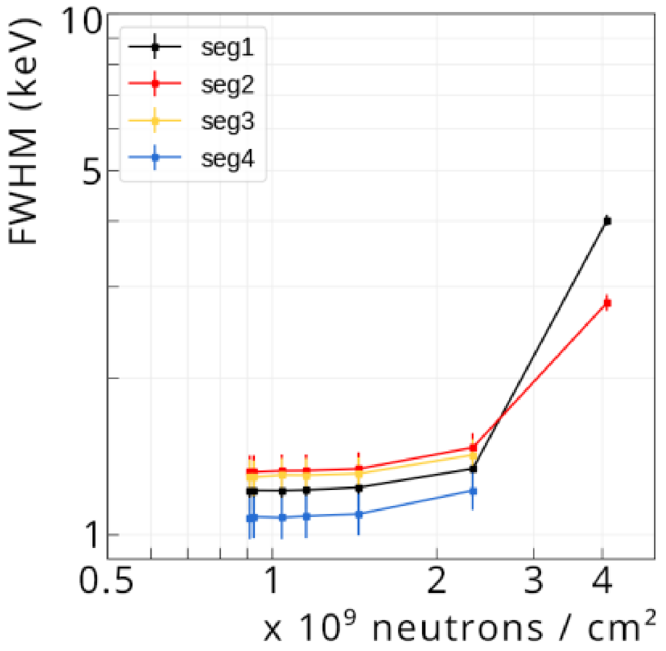
^{60}Co
E = 1332 keV
FWHM < 1,8 keV



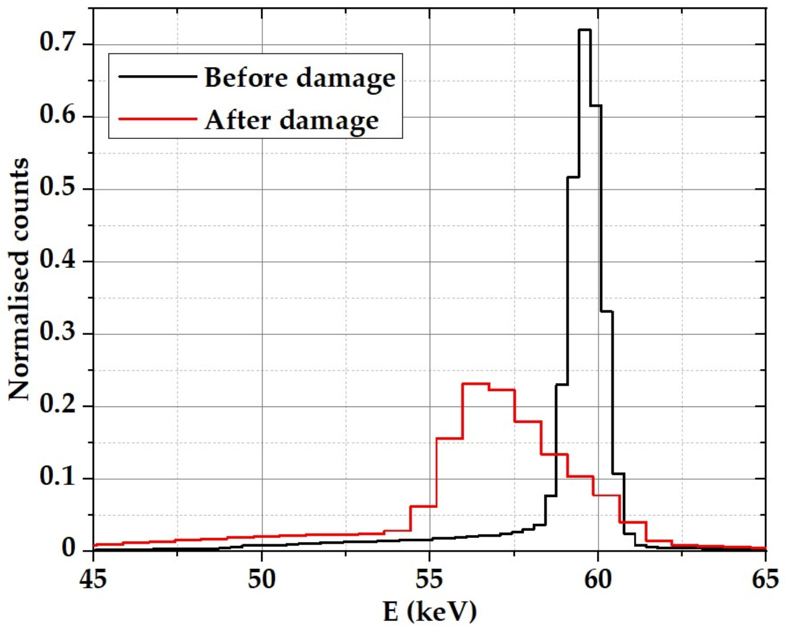
Neutrons damage on planar PLM segmented detector: after 1° run

Operational Voltage 80V
Neutron irradiation for increasing time intervals alternated to 5 min runs with ^{241}Am and ^{60}Co leads to increasing resolution worsening

After 4 hours of irradiation time,
≈ $4{\cdot}10^9$ neutrons/cm^2, detector is no longer operable



^{241}Am
E = 59,5 keV
FWHM = 3,2 – 4,2 keV

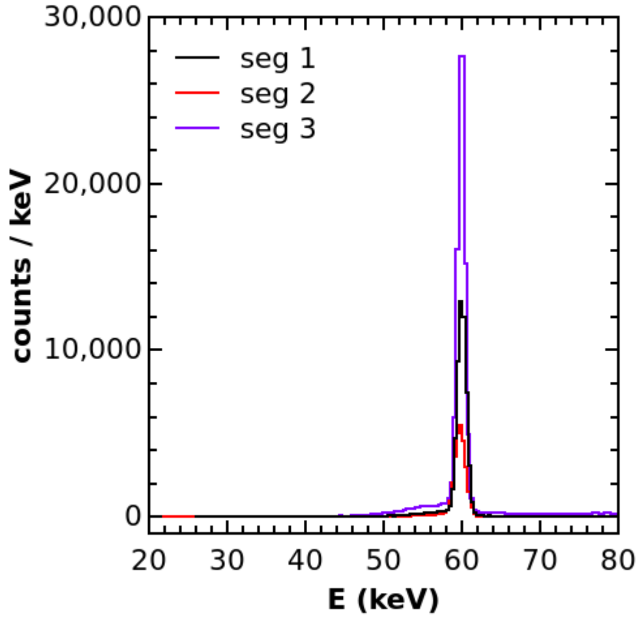


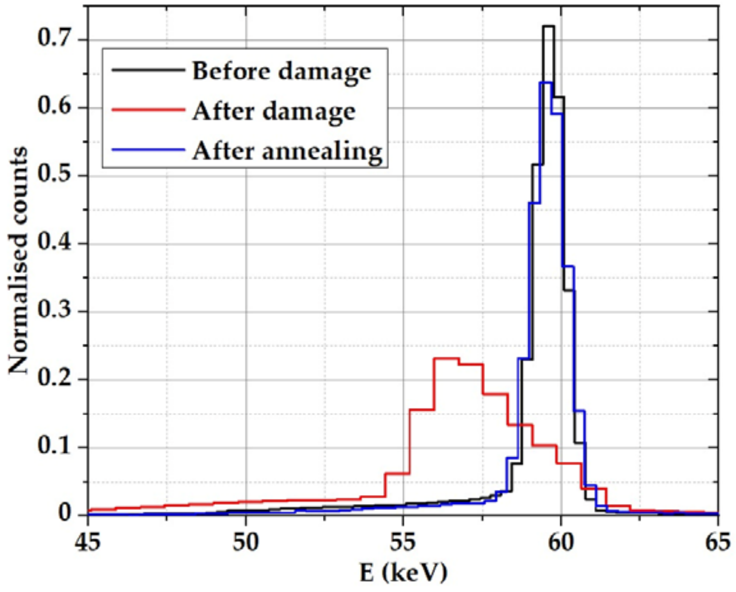
Neutrons damage on planar PLM segmented detector: After Recovery

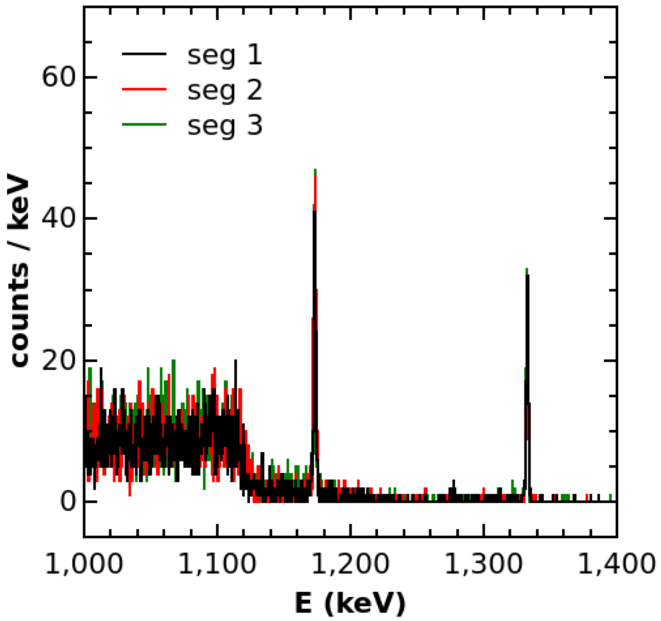
Annealing procedure: 7 days at 105°C continuously pumped inside the cryostat

^{241}Am
E = 59,5 keV
FWHM < 1,6 keV

^{60}Co
E = 1332 keV
FWHM < 2 keV





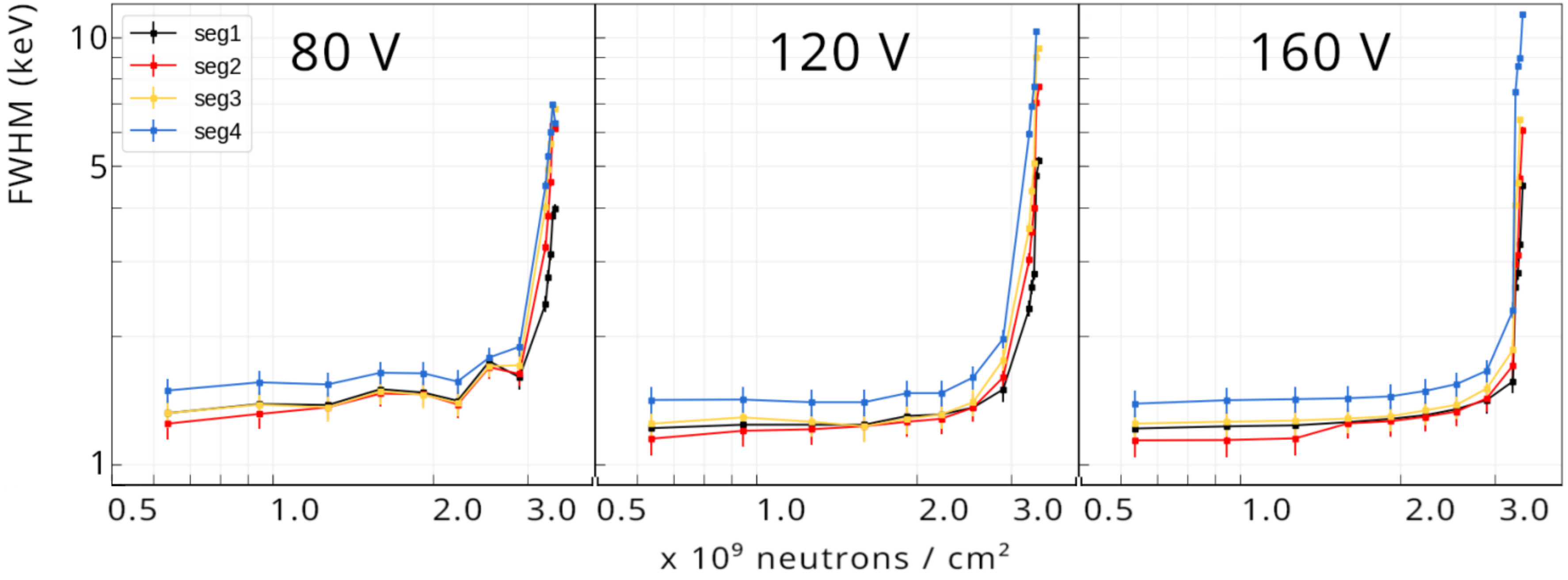


Neutrons damage on planar PLM segmented detector: After 2° run

Operational Voltage 80-120-160V
Neutron irradiation for 20 and 2 min to 5 min runs with ^{241}Am to better characterize resolution worsening

Drastic drop in resolution after $\approx 3\cdot10^9$ neutrons/cm^2 irradiation fluence

^{241}Am
E = 59,5 keV
FWHM = < 2 keV until threshold



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SUMMARY

- PLM technology can be apply to HPGe crystal (hyperpurity preserve)
- PLM junction is thin, segmentable and termally stable (anneling recovery)
- PLM and segmentation technology can be applied to both planar and coxial detectors (2D to 3D shape)
- The HPGe crystal surface preparation and the electrical contact force are fondamental
- PLM segmented detector recovers after Neutron damage with a very good energy resolution

R&D Gamma ray detector Team

Davide De Salvador
Stefano Bertoldo
Enrico Napolitani
Francesco Sgarbossa

Sara Carturan
Gianluigi Maggioni
Francesco Recchia
Dino Bazzacco

Walter Raniero
Daniel Napoli
Chiara Carraro

Stefano Capra
Giacomo Secci
Alberto Pullia
Bénédicte Million
Luciano Manara

Andrea Mazzolari
Lorenzo Malagutti

Andres Gadea

800 ANNI 1222·2022 — Università degli Studi di Padova

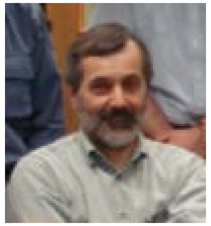
Università degli Studi di Milano

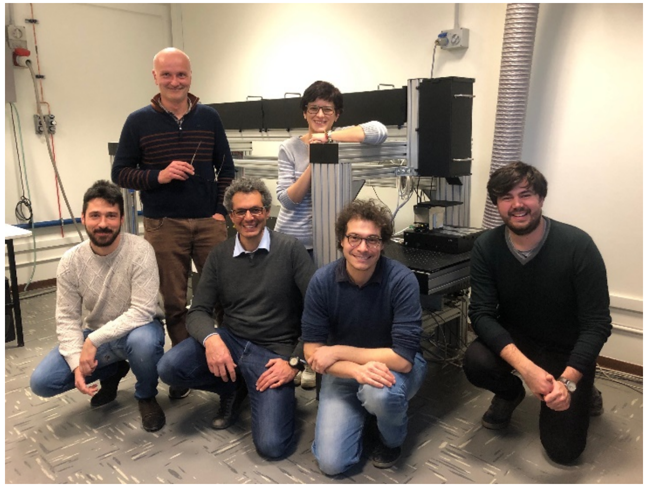
Università degli Studi di Ferrara

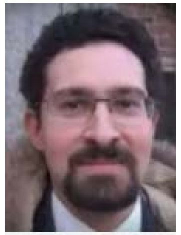
IFIC Institut de Física Corpuscular

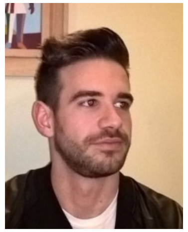
CSIC Consejo Superior de Investigaciones Científicas

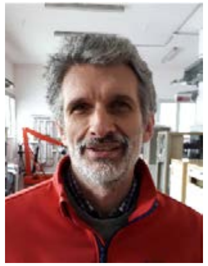
INFN Istituto Nazionale di Fisica Nucleare











InTraNS Gamma Detectors Hands-on Training

LNL-INFN (Italy), 2 - 6 of September 2024

EURO-LABS
EUROPEAN LABORATORIES FOR ACCELERATOR BASED SCIENCES

InTraNS
Instrumentation and Training for Accelerators Based Nuclear Spectroscopy and Reaction Dynamics

Development and test of new technologies for manufacturing high purity germanium segmented detector

Walter Raniero
INFN – LNL

mail: walter.raniero@lnl.infn.it

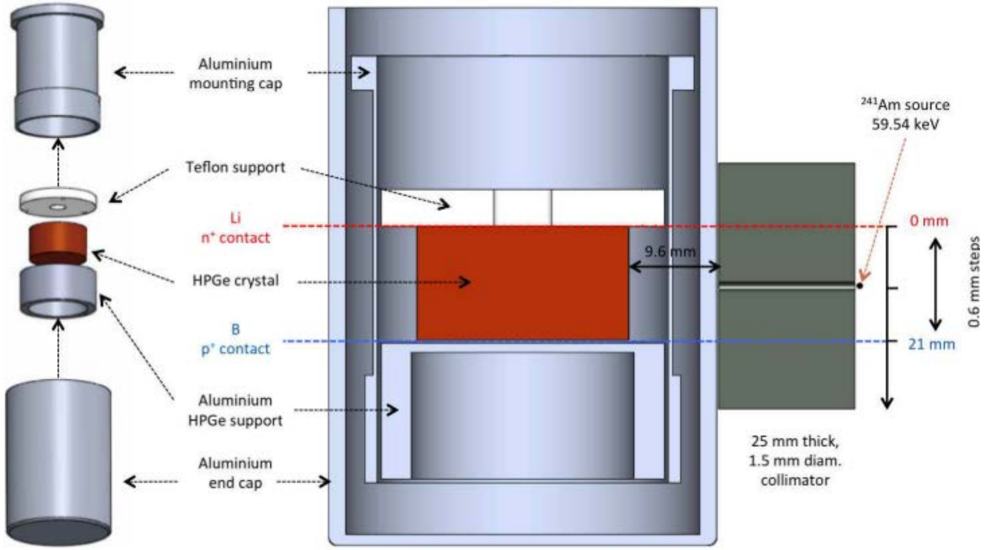
INFN
Istituto Nazionale di Fisica Nucleare

Germanium material for gamma detector (passivation ϒ-ray scanning)

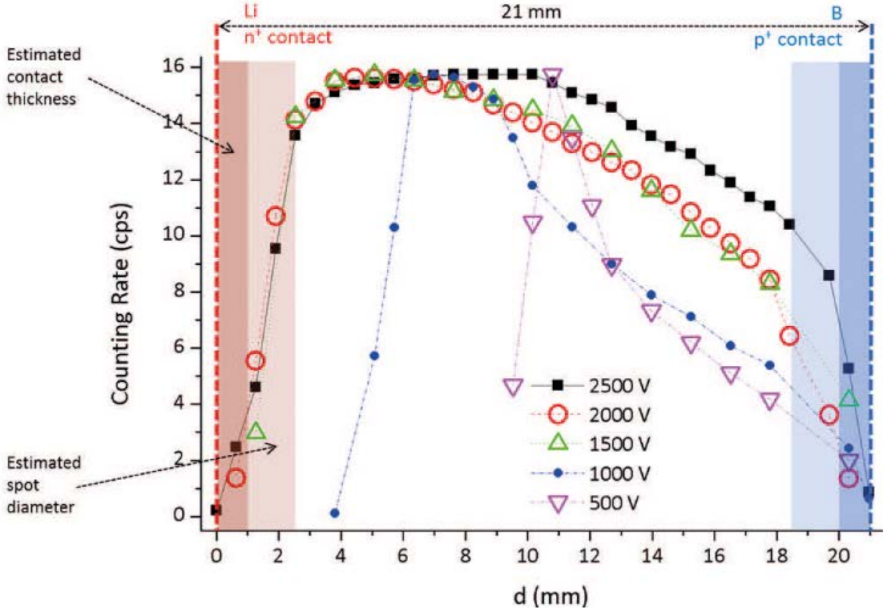
HPGe Passivation: lateral scan ^{241}Am on passivated surface

Cryogenic cryostat
80 ÷ 90K

gamma source ^{241}Am



Depletion Voltage

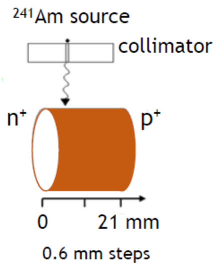


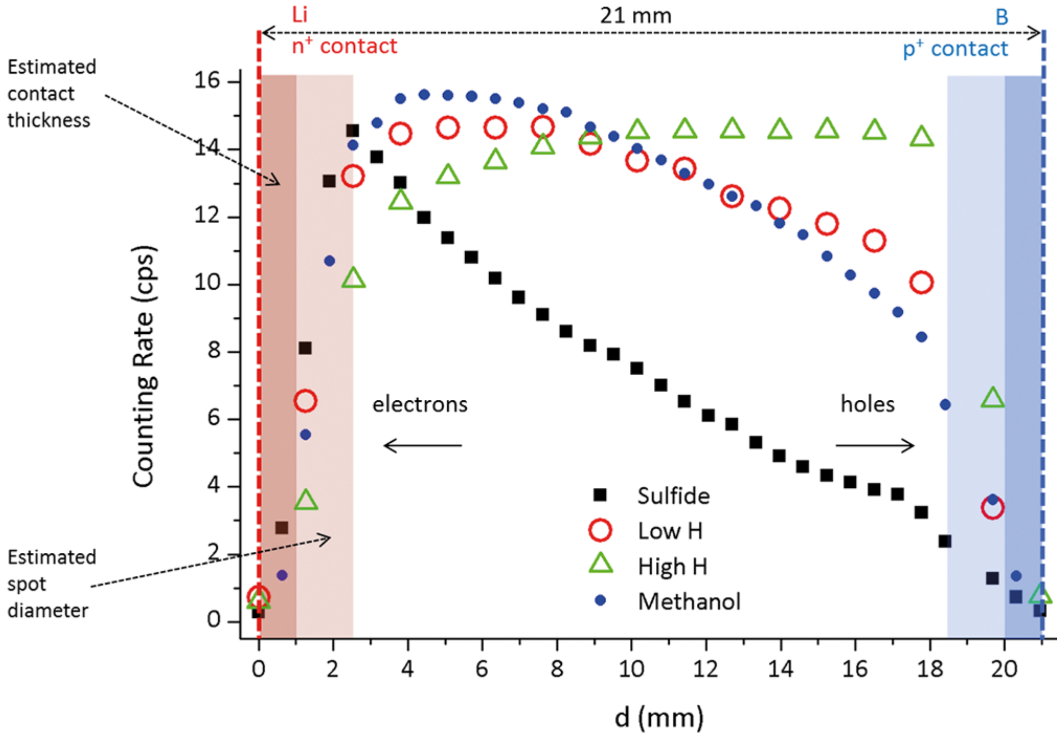
Counting rate (59.5 keV) of the methanol-passivated detector, at differtent voltage apply

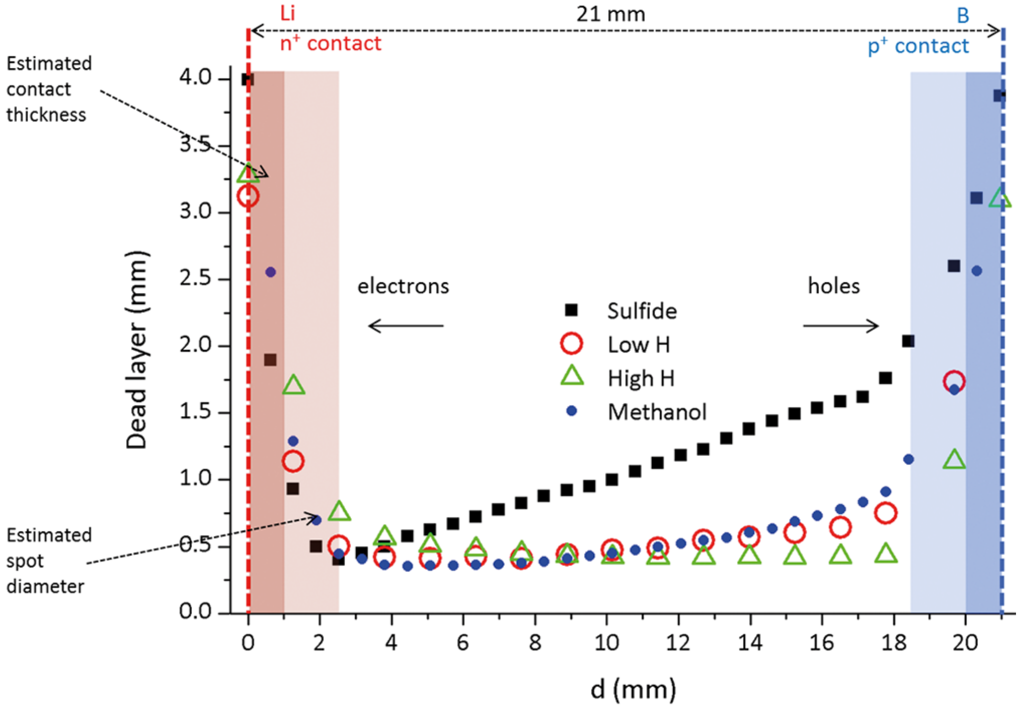
G. Maggioni et al. **Eur. Phys. J. A** (2015) 51: 141

Germanium material for gamma detector (passivation Υ-ray scanning)

HPGe Passivation: lateral scan ^{241}Am on passivated surface







- Methanol termination
- Sulphide termination
- Low Hydride termination (10% HF)
- High Hidride termination (50% HF)

G. Maggioni et al. **Eur. Phys. J. A** (2015) 51: 141

Thin dead layer on passivation surface

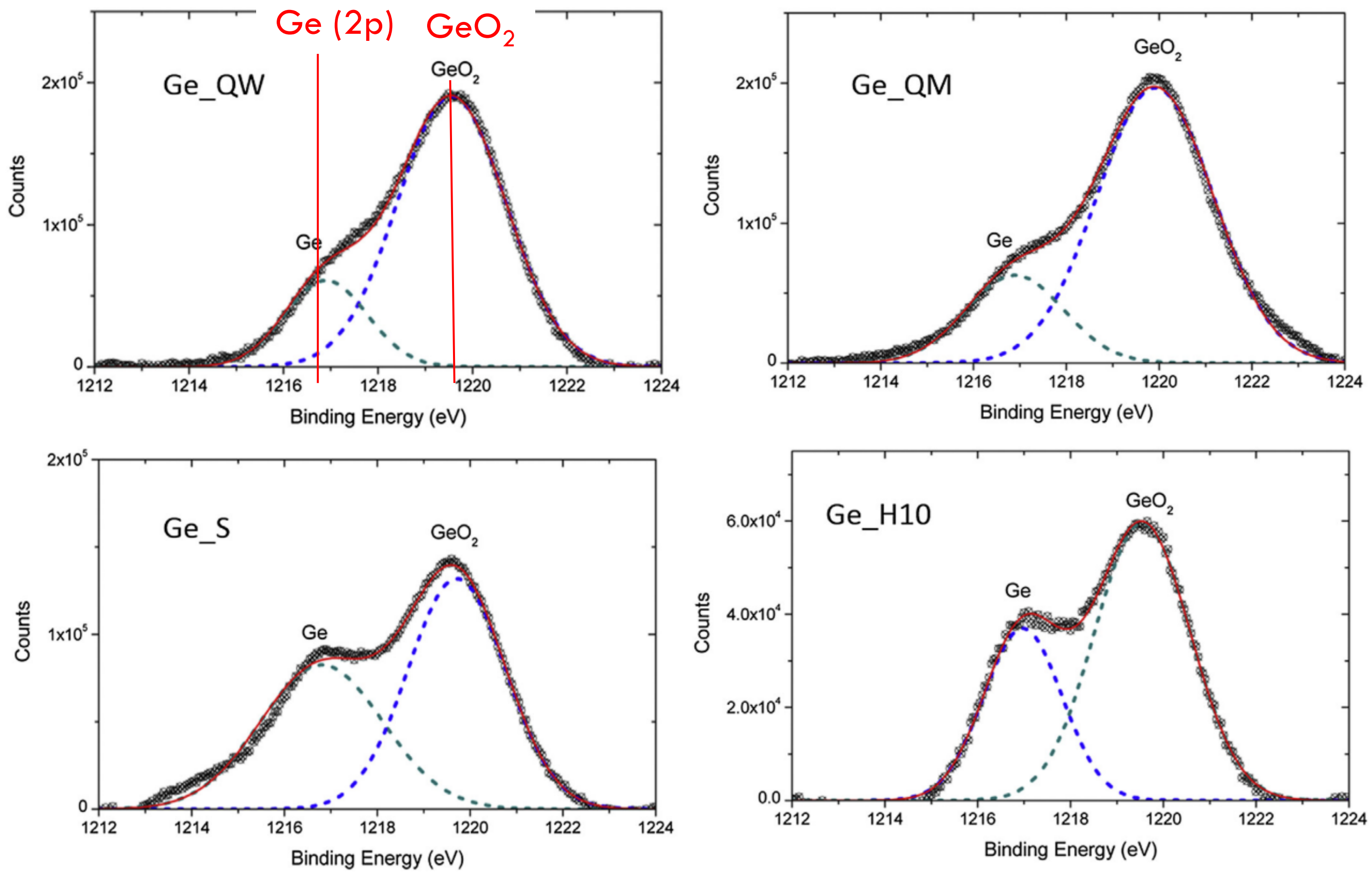
HPGe gamma detector (chemical Passivation)

Passivation techniques: study the evolution of Ge – GeO – GeO_2

Lateral surface passivated

XPS Binding Energy collected after 1 month of air exposure (RH about 55%)

X-ray Photoelectron Spectroscopy (XPS)



S. Carturan et al., **Mater. Chem. Phys.** 2015

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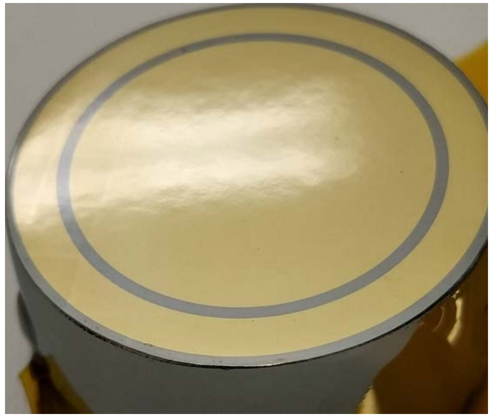
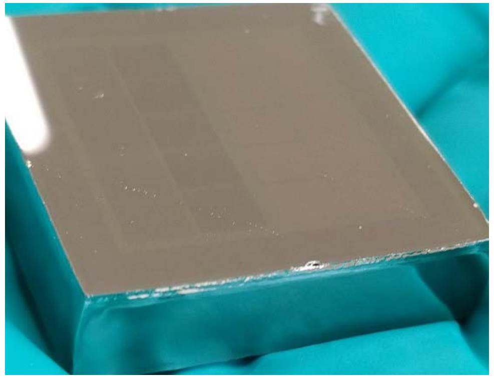
Thick planar HPGe detectors

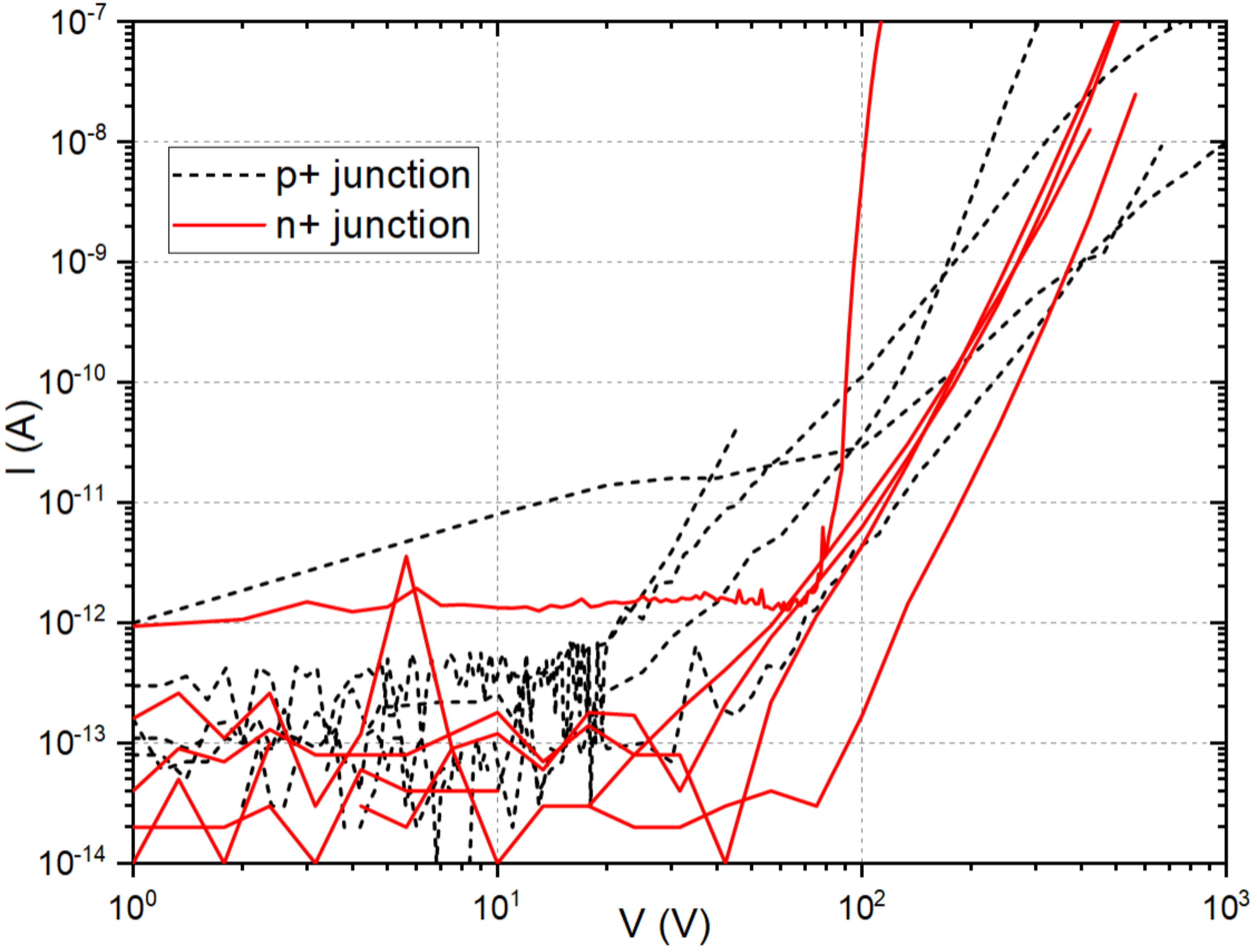
Al/n-HPGE/Sb,
D=40mm, t=20mm



Sb/p-HPGE/Al,
L=35mm, t=10mm

P/p-HPGE/Al, L=35mm,
t=10mm





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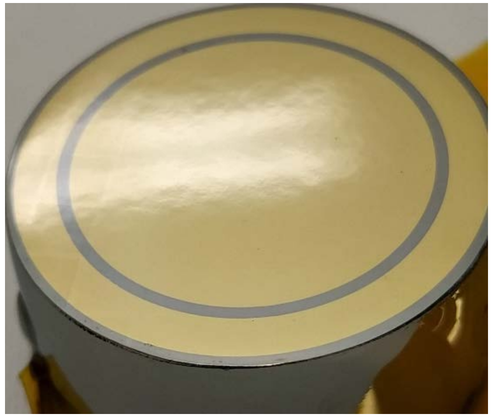
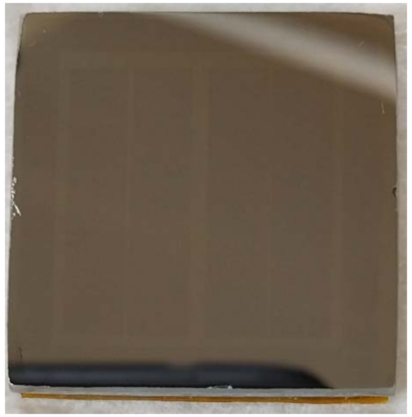
Thick planar HPGe detectors

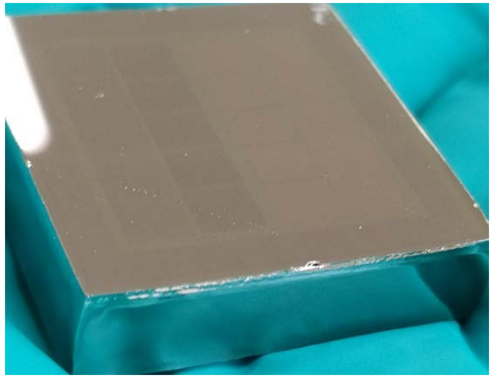
Al/n-HPGE/Sb,
D=40mm, t=20mm



Sb/p-HPGE/Al,
L=35mm, t=10mm

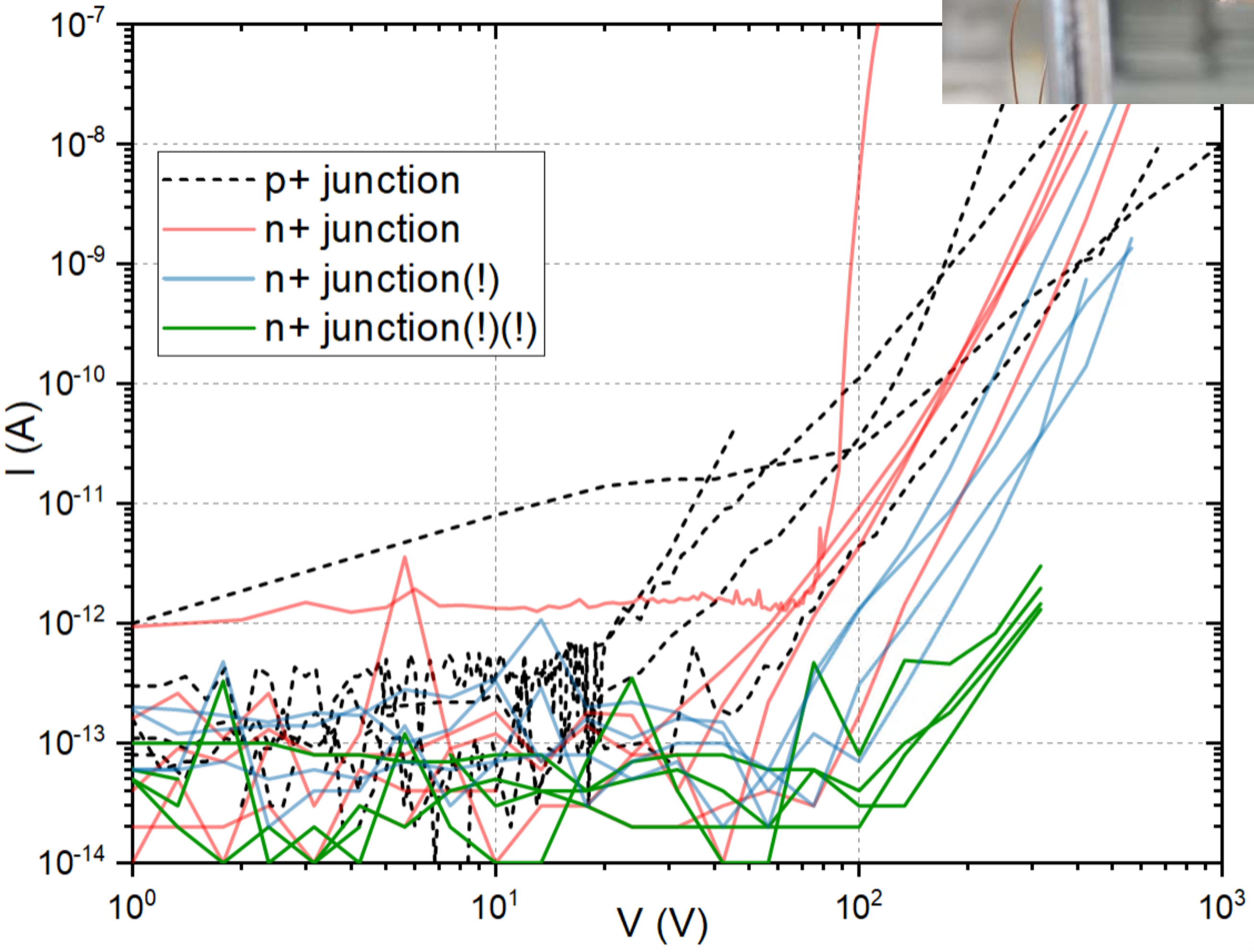


P/p-HPGE/Al,
L=35mm, t=10mm



Difficult to reach depletion voltages (600V for 1cm, 2200V for 2 cm)

Probably due to junction mechanical weakness under manipulation



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